October 1958 Pages 677-732

The Journal of the Society of Dyers and Colourists

Volume 74 Number 10

CONTENTS

PROCEEDINGS OF THE SOCIETY

Some Recent Developments in Dyeing for the Carpet Trade (*W. Beal*) 677

COMMUNICATIONS

Adsorption at Organic Surfaces

IV— Adsorption of Sulphonated Azo Dyes by Chitin from Aqueous Solution (*C. H. Giles, A. S. A. Hassan, and R. V. R. Subramanian*) 682

The Dyeing of Cellulose Acetate with Disperse Dyes

VII— A Comparison with their Solubility in Organic Solvents (*C. L. Bird*) 688

Unlevel Dyeing in Wool Velour Cloth (*F. J. Parker*) 693

NOTES 696

NEW BOOKS AND PUBLICATIONS 700

ABSTRACTS 703

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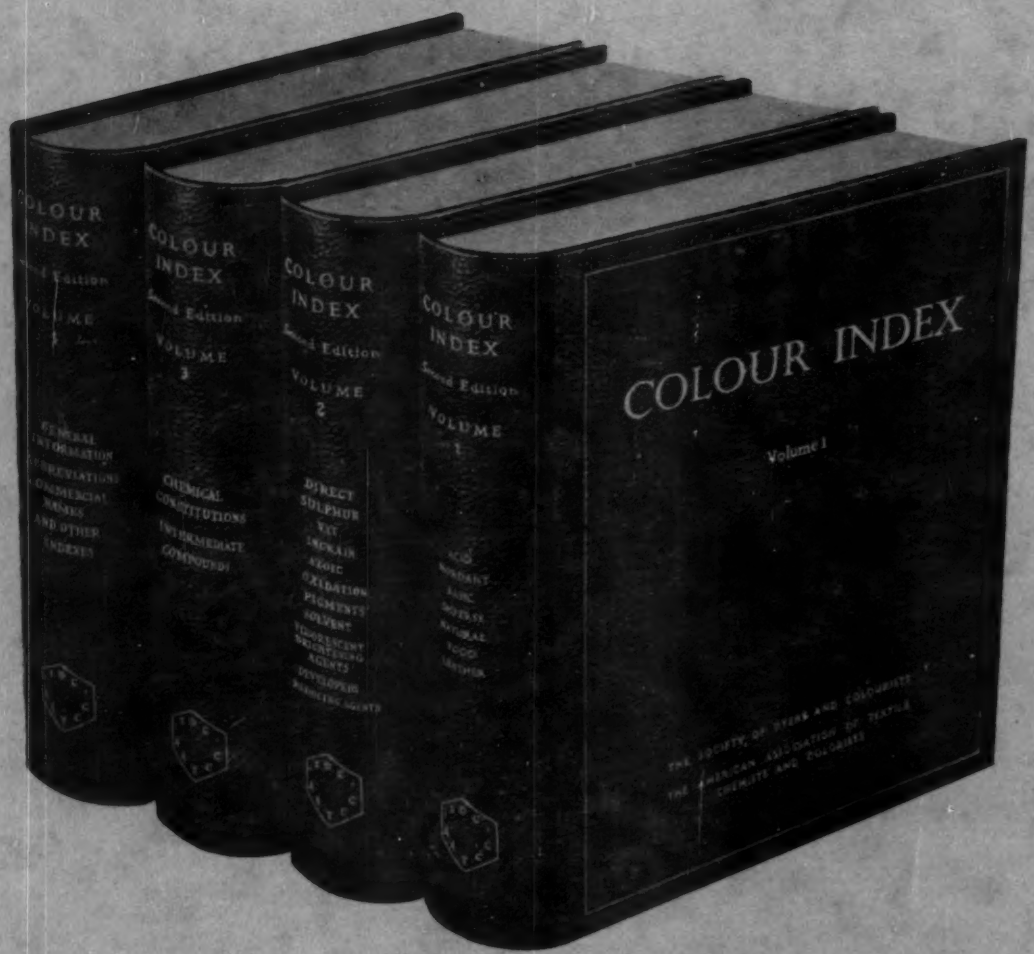

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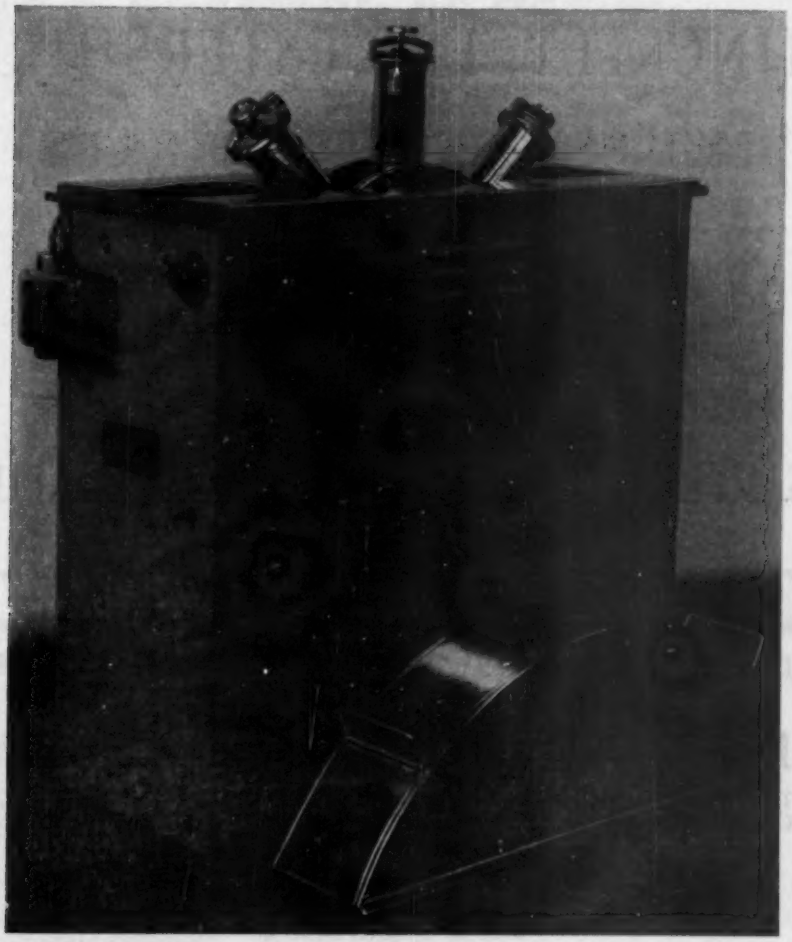

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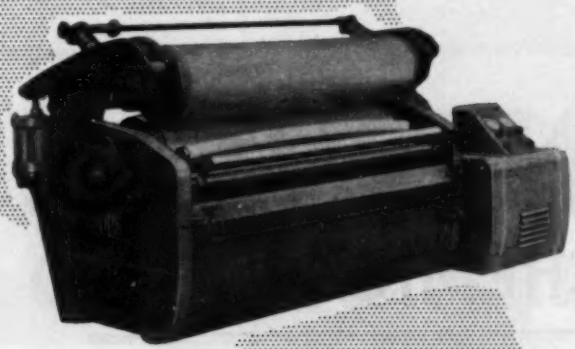

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1–7 of the January 1958 and pages 509–516 of the July 1958 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Determination of Damage to Wool Fibres — *J. C. Brown*

Colour in Writing and Copying — *J. P. Gill*

The Continuous Dyeing of Wool — *D. R. Lemin*

Water-repellent Finishes—Modern Use of Silicones — *G. W. Madaras*

Flameproofing of Textile Fabrics with particular reference to the Function of Antimony Compounds — *N. J. Read and E. G. Heighway-Bury*

COMMUNICATIONS

Reclamation of Fibres from Rags—
III—Action of Hydrazine on Terylene — *A. Filson and J. B. Speakman*

Adsorption at Organic Surfaces—
V—A Comparison of the Adsorption of Solutes by Cellulose and Chitin — *C. H. Giles and A. S. A. Hassan*

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption — *C. H. Giles*

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FORTHCOMING MEETINGS OF THE SOCIETY

Wednesday, 22nd October 1958

BRADFORD JUNIOR BRANCH. *The Properties of Fibres in Relation to Chemical Structure.* Dr. A. Sharples (British Rayon Research Association). Institute of Technology, **Bradford.** 7.15 p.m.

Thursday, 23rd October 1958

WEST RIDING SECTION. *The Solubility of Wool Dyes— Its Assessment and Practical Significance.* W. Beal, Esq., B.Sc. (The Geigy Co. Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 24th October 1958

LONDON SECTION. The Sixth London Lecture. *Colour in Art.* A. Lucas, Esq. (Chief restorer to the National Gallery). The Waldorf Hotel, **London W.C.2.** 7 p.m.

NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

Tuesday, 28th October 1958

LEEDS JUNIOR BRANCH. *Developments in Modern Dry Cleaning Techniques.* E. J. Davies, Esq., M.Sc. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

Wednesday, 29th October 1958

BRADFORD JUNIOR BRANCH. Visit to Stevensons (Dyers) Ltd., **Ambergate.**

MIDLANDS SECTION. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. King's Head Hotel, **Loughborough.** 7 p.m.

Wednesday, 5th November 1958

The Fifth George Douglas Lecture on *Finishing.* Dr. Walter Gutmann (Farbwerke Hoechst AG.). The Midland Hotel, **Bradford.** 7.15 p.m.

Thursday, 6th November 1958

BRADFORD JUNIOR BRANCH. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

Friday, 7th November 1958

LONDON SECTION. Investiture of the Chairman's Badge by the President. *Some Aspects of the Dyeing and Finishing of Fabrics Knitted from Bulky and Stretched Yarns.* S. M. Jaeckel, Esq., B.Sc., A.R.C.S., A.R.I.C., F.C.S., and D. L. Munden, Esq., B.Sc. (The Hosiery & Allied Trades Research Assoc.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

WEST RIDING SECTION. ONE DAY CONFERENCE.
Dyeing Unions containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.).
Dyeing of Polyester Fibres and Blends. H. W. Partridge, Esq., M.Sc., A.T.I. (Stevensons (Dyers) Ltd.).
Finishing of Blended Fabrics containing Wool and Acrylic or Polyester Fibres. C. S. Whewell, Esq., B.Sc., Ph.D., F.R.I.C., F.T.I., F.S.D.C. (Leeds University).
The Uses of Acrylic Fibre Blends with reference to Colour Fastness. H. D. Edwards, Esq., B.Sc., Ph.D., A.R.I.C. (Courtaulds Ltd.).
Institute of Technology, **Bradford.** 2 p.m.

Tuesday, 11th November 1958

LEEDS JUNIOR BRANCH. *Theoretical Aspects of Solution Chromatography.* Dr. A. Levi (Imperial Chemical Industries Ltd.). Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

NORTHERN IRELAND SECTION. *General Principles in Pigment Padding with Vat Dyes.* Dr. R. Kern. (Sandoz, Basle). Grosvenor Rooms, **Belfast.** 7.30 p.m.

Thursday, 13th November 1958

WEST RIDING SECTION. *The Appearance of Textile Faults in Dry Cleaning.* E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation.) Griffin Hotel, Boar Lane, **Leeds.** 7.30 p.m.

Tuesday, 18th November 1958

HUDDERSFIELD SECTION. *The Dry Cleaning of Textiles.* E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation). Silvios Cafe, **Huddersfield.** 7.30 p.m.

MIDLANDS SECTION. *The use of Anti-static Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Joint meeting with the Nottingham Textile Society.) Gas Board Theatre, **Nottingham.** 7 p.m.

Tuesday, 18th November 1958

SCOTTISH SECTION. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 19th November 1958

SCOTTISH JUNIOR BRANCH. *Some Textile Dyeing and Drying Machines.* K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). (This lecture will be illustrated by means of a film show.) Technical College, George Street, **Paisley.** 7.30 p.m.

Thursday, 20th November 1958

LONDON SECTION. *Developments in Modern Dry Cleaning Techniques.* E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation). (Joint meeting with the London Centre of the Guild of Dyers & Cleaners.) Institute of Journalists, 2-4 Tudor Street, **London E.C.4.** 7.15 p.m.

MANCHESTER JUNIOR BRANCH. *Textile Microscopy.* G. Cartridge, Esq. Room J/B 16—Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 21st November 1958

BRADFORD JUNIOR BRANCH. *The Dyeing of Blacks, Navy Blues and other Heavy Shades on Tricel by Azoic Methods.* W. R. Beath, Esq., M.Sc., A.M.C.T. and J. S. Ward, Esq., B.Sc. (Courtaulds Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

HUDDERSFIELD SECTION. Annual Dinner. Princess Cafe, Northumberland Street, **Huddersfield.**

MANCHESTER SECTION. Ladies Evening. College of Science and Technology, **Manchester.** Further details later.

Monday, 24th November 1958

HUDDERSFIELD SECTION. Invitation from The Halifax Textile Society— Joint meeting at The Alexandra Hall, **Halifax.** 7.30 p.m.

Thursday, 27th November 1958

WEST RIDING SECTION. *Photoelectric Colorimeters— Their Uses and Limitations in the Dyehouse.* J. V. Alderson, Esq., B.Sc., A.R.C.S. (Imperial Chemical Industries Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Tuesday, 2nd December 1958

LEEDS JUNIOR BRANCH. *The Dyeing of Wool-Fibro Blends.* Miss E. Smith. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

SCOTTISH JUNIOR BRANCH. *Some of the Principles and Practices of Textile Printing.* A. Howarth, Esq., A.M.C.T., F.S.D.C., A.T.I. (The Seedhill Dyeing & Printing Co., Ltd.). Technical College, George Street, **Paisley.** 7.30 p.m.

Friday, 5th December 1958

BRADFORD JUNIOR BRANCH. Diamond Jubilee Dinner Dance.

LONDON SECTION. *Dyeing of Furs.* J. L. Stoves, Esq., M.Sc., Ph.D., F.R.I.C., F.C.S. (C. W. Martin & Sons Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 9th December 1958

BRADFORD JUNIOR BRANCH. *The Continuous Dyeing of Synthetic Fibre Blends.* D. A. Garrett, Esq., B.A., A.Inst.P. and P. J. Dolby, Esq., B.Sc., F.C.S. Institute of Technology, **Bradford.** 7.15 p.m.

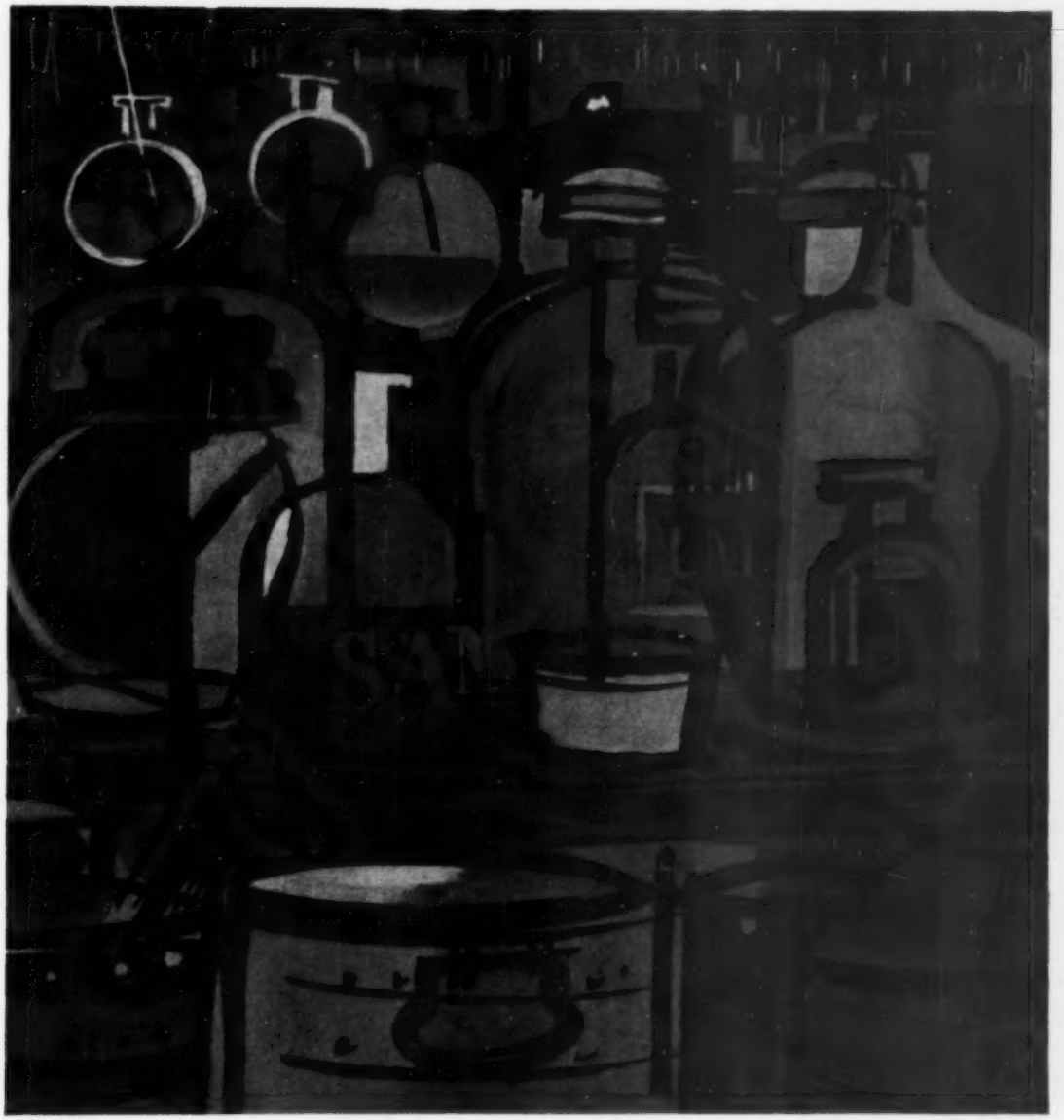
NORTHERN IRELAND SECTION. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R I C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Fast Shades on Wool-Cellulose Unions.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Wednesday, 10th December 1958

MIDLANDS SECTION. (Title and Lecturer to be announced later.) King's Head Hotel, **Loughborough.** 7 p.m.

continued on page xii



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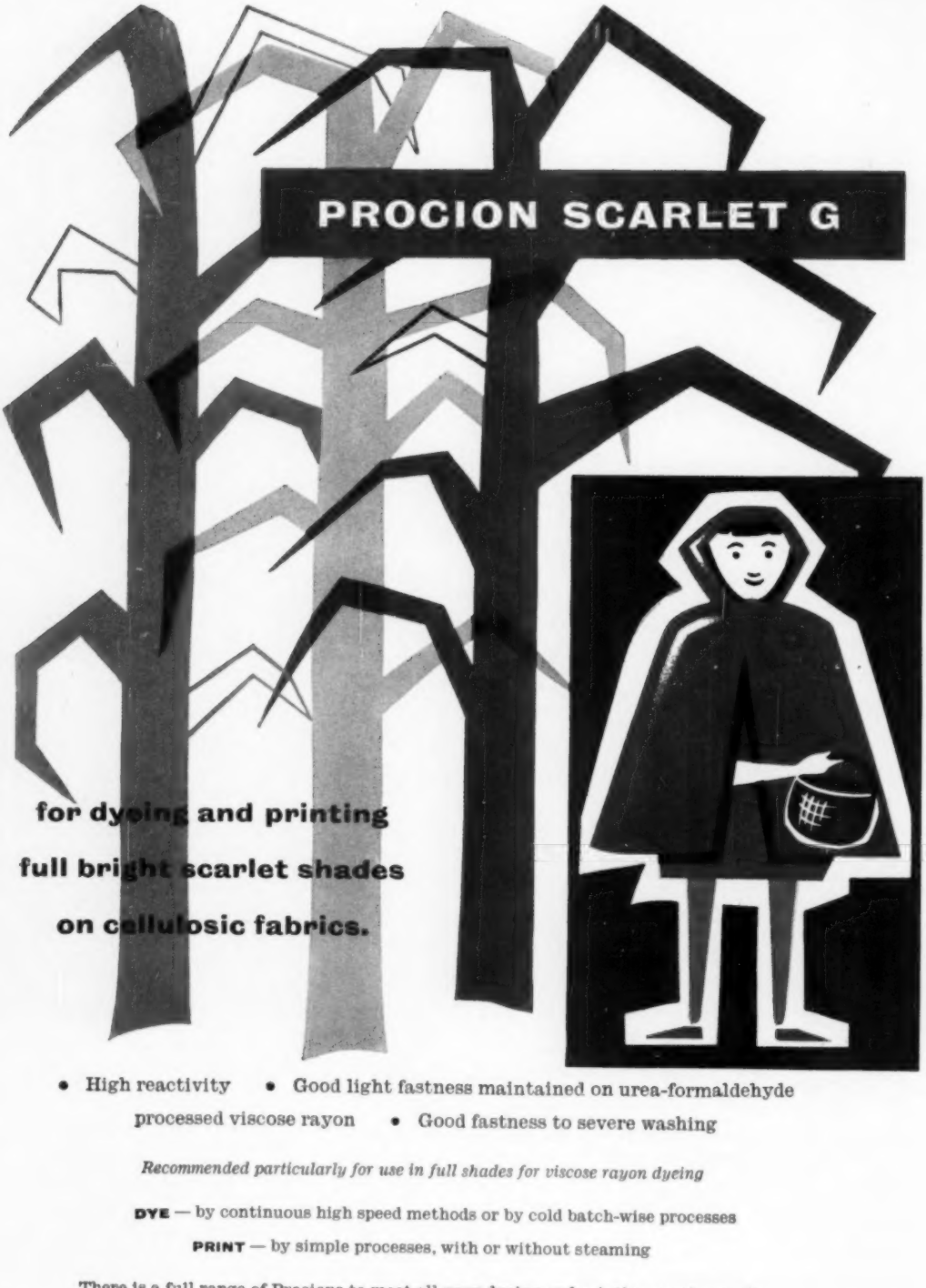

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 74 Number 10 OCTOBER 1958 *Issued Monthly*

Proceedings of the Society

Some Recent Developments in Dyeing for the Carpet Trade

W. Beal

Meeting of the Midlands Section held jointly with the Kidderminster Section of the Textile Institute at Messrs. Carpet Trades Ltd. Canteen, Mill Street, Kidderminster, on 3rd April 1958, Mr. W. C. F. Swindell in the chair

The large-scale introduction of synthetic fibres into the carpet trade has caused many dyeing problems. The way in which these problems are being overcome is reviewed together with developments which have been taking place in dyeing for all-wool pile carpets.

Introduction

For many years there was little change in the types of dye used for the carpet industry, but in the past few years considerable advances have been made. We have seen the rapid development of the tufted carpet, and this has created new fields for itself as well as competing to some extent with the traditional woven carpet. Tufted carpets were initially made from a pile yarn of 100% viscose rayon staple, and this helped to give them a very attractive price. Woven-carpet manufacturers were thus at a considerable disadvantage when using an all-wool pile yarn, and consequently we have had the development of the union carpets, which have utilised some of the cheaper synthetics.

In addition, we have seen the remarkable growth of the range of synthetic fibres, which has given manufacturers the chance of producing new effects, but has also given the dyer many problems. Against this background, it is quite understandable that many dyers have had to experiment with new ideas and new techniques, and, at least to some extent, the dye manufacturers have been able to give some assistance in the initial stages.

Woven Carpets

ALL-WOOL YARNS

So far as conventional woven carpets are concerned, the all-wool pile carpet is still the most important, but even in this field several important changes are occurring. The customer is becoming more fastness-conscious, and to meet his demands new types of dye have been introduced for carpet yarn dyeing. Full reds have been very popular for several years, and where previously these have been dyed with Carmoisine (C.I. Acid Red 14). dyes of improved water fastness are now being used to a large extent, e.g. Naphtol Red B (C.I. Acid Red 27), which has similar dyeing characteristics to Carmoisine, and Polar Reds such as Polar Red RS conc. (C.I. Acid Red 99) and Polar Brilliant Red 10B conc. (C.I. Acid Violet 54). This latter group is more difficult to apply, since the dyes have much lower migration, but they do give the maximum fastness to wet treatment whilst maintaining a satisfactory standard of light fastness.

Another change that is occurring at the moment is the replacement of the traditional level-dyeing acid blue, of the Erio Fast Cyanine S conc. (C.I. Acid Blue 45) type, by a blue which is much less sensitive to salt spotting or urine, e.g. Erio Anthracene Brilliant Blue F3GL (C.I. Acid Blue 72), which is equally level-dyeing. This new type of blue is somewhat more expensive than the previous type, but it does ensure freedom from complaints, which have been increasing in numbers, probably owing to the prevalence of charcoal greys in present-day designs.

Particularly for the export trade, pale colours have been very popular, and this has demonstrated quite clearly the deficiency in light fastness of the three-colour level-dyeing acid-dye combination. The neutral-dyeing metal-complex dyes of the Irgalan type have extremely good light fastness in pale colours, and for this reason they are being introduced in ever increasing quantities for carpet-yarn dyeing. For a long while it was thought that dyes of this type could be satisfactorily applied only on loose wool, and this was a severe limitation to their usefulness, but for some considerable time now there has been no doubt of their ability to be applied satisfactorily to carpet yarn. The fastness properties of the metal-complex dyes are generally considered to be satisfactory in every respect, and for pale to medium depths the extra cost is of little consequence.

These dyes also give a better coverage of the various blends used in carpet yarns and thus result in an improved appearance. Sometimes it is possible to save money by using a cheaper blend and dye with Irgalans to produce the same overall coverage as was previously obtained by using conventional dyes on a more expensive blend. For example, one firm had always paid a few pence extra to make a better blend when dyeing greens in order to obtain the same coverage as with the other colours in their range. This had always been necessary because of the poor coverage given by the orthodox green dyes previously available. With the introduction of Irgalan Brilliant Green 3GL they discovered that excellent coverage could be obtained on their normal blend by using this metal-complex dye, and the difference in cost between the two types of dye was far less than the difference in cost between the two blends.

In addition, one must not forget the ease of dyeing, particularly on loose wool, of the metal-complex dyes and the fact that a considerable saving in time and steam, etc. is afforded by their short dyeing time and the reproducibility of the results obtained. Using this type of dye, it is also possible to develop new dyeing methods, e.g. continuous dyeing, and this point is discussed in detail on p. 681.

MIXTURE YARNS

We have two important mixture yarns to consider, viz. wool–viscose rayon staple and wool–secondary cellulose acetate. Of these, the former carpet yarns have been used to a much greater extent than all the other mixtures combined [1].

Wool–Viscose Rayon

The great advantage of wool–Fibro yarns is that the Fibro (Courtaulds viscose rayon staple) component has a stable price of 24 d./lb., and this produces a yarn which is attractive in price. It is hard wearing and can be made to look very attractive, but it has the drawbacks that it soils more quickly than an all-wool pile and also it has relatively poor resilience [2]. However, there is obviously a great demand for this type of carpeting, and it would appear that almost all the large firms have accepted this position and put out a new range of standard patterns on these lines. Solid dyeings are almost inevitably required, and these necessitate the use of direct cotton dyes and neutral-dyeing wool dyes together in the same dyebath.

Many direct cotton dyes stain wool appreciably, and this staining often has poor fastness. In addition, the degree of staining varies greatly with the temperature, the pH, and the electrolyte content of the dyebath, and it is obviously not easy to control. From all points of view, therefore, it is advisable to choose those direct cotton dyes which give minimum staining of wool, so that the dyeing process can be more accurately controlled. A further selection must be made from such dyes in order to obtain the standard of fastness required on the fibre; and other factors which must not be forgotten are level-dyeing properties, colour, and price. When all these various factors have been examined, one is left with only a very small number of direct cotton dyes which are suitable, and this number is further limited where the electrolyte content of the water is very high. Sometimes the Fibro used is insufficiently desulphurised, in which case the metal may be removed from some of the direct cotton dyes which are metal complexes, and this results in a change of colour and lower light fastness. Finally, we must ensure that the dyes chosen are satisfactory in combination, as this is by no means certain, even though each dye may be individually satisfactory.

It is therefore not surprising that many firms have chosen to purchase ready-made union dyes rather than to try and use separate wool and cotton dyes to produce solid dyeings on wool–Fibro yarns, and ranges of union dyes of various grades of fastness and, incidentally, of price are available. Cheaper dyes such as Uniphenyl and Uniphenyl Fast are chiefly used for medium to full depths, where their fastness is adequate and where the cross staining is not particularly troublesome. For pale to medium depths, dyes of a much higher fastness to light are required, and ranges such as Union Fast SL have been developed to meet these demands.

Many firms have found, on going into this form of dyeing for the first time, that they have had to change their previously held ideas very considerably. A common practice is to produce the majority of colours on all-wool yarn with a combination of red, yellow, and blue dyes, and this is perfectly satisfactory when level-dyeing acid dyes are used. With dyes of limited migration, however, such as are used in these union mixtures, this method of building up colours is seldom satisfactory, as skittery or two-tone dyeings are often produced [3]. It is preferable to select a dye with a colour which is reasonably similar to that which has to be produced, and to shade slightly as required in order to prevent these faults from occurring. Thus if a grey has to be dyed, one should start from a grey dye; or if a brown is required, then a brown should be used as a basis. This puts an added strain on the dye warehouse, but it is desirable if the dyer is to produce satisfactory results.

Another important point is control of pH during dyeing, as even the best direct dyes tend to give some staining of wool under acid conditions, and many of the wool dyes used in such unions yield level dyeings only under relatively neutral conditions. On the other hand, if the dyebath is allowed to become too alkaline most of the wool dyes will not exhaust satisfactorily, and there is a strong tendency for many of the direct cotton dyes to be reduced or hydrolysed. Accurate pH control is therefore required, and this is something that is relatively new to the majority of carpet-yarn dyers, who previously have not had to worry unduly about the pH of the yarn as it comes forward for dyeing or that of the water which is being used, since these are of relatively little consequence in acid dyeing. This pH control must allow for the increase in alkalinity developed at high

temperatures by water which contains bicarbonates, which are converted to the corresponding carbonates as the temperature of the dyebath is raised.

It is also necessary to have more accurate control of temperature than is commonly used in applying the level-dyeing acid dyes to wool, since a high temperature favours the dyeing of wool and a low temperature the dyeing of Fibro. The ideal temperature for dyeing is of the order of 190–195°F., and as a general rule boiling should not be allowed to take place, since this greatly increases the risk of destruction of the direct cotton dye by hydrolysis or reduction, and this has been the cause of many unfortunate experiences of dyers in this field.

One final point of importance is correction of the hardness of the water to avoid dusting of the yarn [4]. A metal-sequestering agent which is stable at temperatures up to the boil is required, e.g. ethylenediaminetetra-acetic acid (E.D.T.A.) compounds such as Irgalon BT, a concentration of 4 oz. per 100 gal. per 10° hardness giving the desired result. The inorganic phosphates are not suitable for this purpose, since they decompose at high temperatures. When E.D.T.A. compounds are used it is essential to ensure that the dyes themselves are unaffected. Many of the direct cotton and wool dyes used in yarn dyeing are metal-complex dyes, and it is essential to ensure that the E.D.T.A. compound used will not affect the stability of the dye molecule. If the metal is removed from the complex, the colour and fastness properties of the dyeing are greatly affected.

Similar problems exist in the dyeing of Fibro–Fibrolane mixtures, which, although employed in the trade to only a small extent at the moment, seem to be on the increase [5]. Fibrolane (Courtaulds' regenerated protein fibre) dyes in a similar manner to wool, but there is a greater degree of cross-staining of the Fibrolane by the direct cotton dye than would be obtained on wool. Because this blend is rather weaker than a wool–Fibro blend, some firms add a third component, e.g. 5–10% nylon. This, of course, means that the dyes have to be selected from yet a further point of view, viz. the necessity of obtaining the same hue and depth of colour on the nylon as on the other fibres.

For several years now we have used another method of dyeing wool–cellulosic fibre unions for the velvet and pile-fabric industry, and we are now examining the possibilities of using it in carpet-yarn dyeing. This method enables wool–cellulosic mixtures to be dyed by a one-bath method under acid conditions, viz. pH 4–5, using acetic or formic acid together with a textile chemical which minimises the staining of the wool by the direct cotton dye, e.g. Erional NW [6].

The advantages of this method compared with the normal dyeing method are—

(1) It gives better exhaustion
(2) It gives better fastness to water and rubbing, mainly because of the greatly decreased cross staining of the wool dye on to the cellulosic fibres
(3) It enables wool to be dyed under ideal pH conditions. This may be especially important if redyeing has to take place or if Fibrolane is used in the blend
(4) It minimises the need for very accurate pH control
(5) It eliminates the problems of alkaline hydrolysis or reduction of the direct dye.

Its disadvantages are—

(1) Only selected dyes can be used, i.e. those direct cotton dyes which can be adequately prevented from dyeing wool
(2) Additional expense is entailed because extra chemicals are needed (approx. 1 d./lb.). This is offset by the saving in dye cost due to the extra exhaustion obtained.

This method gives the greatest advantages in medium to full depths. The final details of the process are still being worked out from the practical trials at present being conducted.

Wool–Secondary Cellulose Acetate

With the second major union used in this field, wool and secondary cellulose acetate, many of the same general problems still apply as with wool and viscose rayon. The acetate has to be dyed with disperse dyes, which as a class tend to give a considerable degree of cross-staining on the wool, and this staining almost invariably has poor fastness to light and rubbing. On the other hand, there is very little staining of the cellulose acetate by wool dyes. Since the disperse dyes are not truly in the solution, they have to be applied in the presence of a dispersing agent, and many of these are available, ranging from soap to anionic and non-ionic synthetic detergents. For the dyeing of wool–cellulose acetate carpet yarns, certain non-ionics, e.g. Irgasol PMC or Tinegal VR, give adequate dispersions and also greatly minimise the cross-staining of the wool by the disperse dye.

The pH is again very important in this form of dyeing, since if the dyebath is allowed to become appreciably acid, the cross-staining of the wool increases very rapidly, but on the other hand, it is not advisable to dye under very alkaline conditions because of the effect on the wool fibre and the exhaustion of the wool dye. It is usual to dye these unions at 185–190°F. in order to minimise the damage to the cellulose acetate fibre, but some improvement in the cross-staining of the wool by the disperse dyes can often be effected by giving a final 10-min. treatment at or near the boil.

Wool–cellulose acetate unions are more expensive than wool–Fibro (secondary cellulose acetate 33 d., Fibro 24 d.), but the wool–acetate carpet is said to have better resistance to soiling and somewhat better resilience.

It will thus be seen that the essential points to be considered when moving from the all-wool to the wool-union field are as follows—

(1) Dye selection
(2) pH control
(3) Temperature control

(4) Type of water to be used
(5) Dyeing assistants required.

In order to obtain satisfactory levelness and penetration the mechanical aspect of dyeing must not be overlooked. It is usually found that, when cellulosic fibres are being dyed, only two-thirds of the weight compared with the corresponding wool yarn can be dyed in the same vessel. In wool–Fibro dyeing one should therefore reduce the weight of yarn dyed in any vessel, compared with a corresponding wool dyeing, by an amount depending on the percentage of Fibro in the blend.

Many other unions have been tried by various manufacturers, but the following have apparently proved of little interest commercially—

(*a*) Wool and cellulose triacetate
(*b*) Wool and acrylic fibre
(*c*) Fibro and acrylic fibre
(*d*) Wool and polyester fibre
(*e*) Wool and nylon.

ACRYLIC-FIBRE YARNS

There is, however, one other type of carpet yarn which is potentially important and which is just finding its feet in this country, viz. 100%-acrylic yarn made from such fibres as Acrilan (Chemstrand) or Courtelle (Courtaulds). These yarns have the advantages of a stable price, warmth, resilience, and very good wearing properties. They are also resistant to attack by moth grubs, carpet beetles, etc. Except for this pest resistance and perhaps the length of service they will give, it is difficult to see where they show to advantage against an all-wool carpet, particularly in view of the fact that they are not cheap fibres. However, they do lend themselves to being used on different types of machinery, such as those used to produce the new types of carpet and the conventional woven type.

Tufted Carpets

In recent years there has been a flood of new types of carpet on the market, and the most important of these are probably the tufted carpets. These carpets can be manufactured at relatively high speeds, but as yet they have been limited to either plain colours or very simple designs. Much research is going on into the problem of producing more complex designs, and various printing methods are being examined.

Tufted carpets may be manufactured from dyed yarn, or piece dyeing may be carried out after making up. The latter method has the advantage that undyed stocks can be built up and quickly dyed to any colour once an order has been placed, but the former has the advantage that a batch of any size can be prepared to exactly the same shade without the attendant worries of piece dyeing. If piece dyeing is to be carried out, the primary consideration in dye selection is level dyeing. When the carpet is made from dyed yarn, which may have been dyed in yarn form or as loose stock, then a wider range of dyes is available.

Until very recently it had apparently been found preferable to use a fibre with a smooth surface for this type of carpet, and hence wool has not normally been used. The main fibre to be used has been Fibro, which is very cheap and hard wearing, but has the disadvantage that it soils very quickly and also has poor resilience. Similar limitations apply to most synthetic fibres, but acrylics present us with new possibilities. They are capable of being used on a tufting machine, but they are warm to the touch, relatively resilient, and extremely hard wearing, although it is true that they soil more quickly than wool. Pale colours can be produced on all the acrylic fibres with disperse dyes of the Setacyl type, but some acrylics such as Orlon (DuP) and Courtelle have insufficient affinity for these dyes for them to be used in medium to full depths.

Two different types of acrylic fibre [7] are used—

(1) Copolymer of acrylonitrile with a basic substance, e.g. Acrilan
(2) Polymer of acrylonitrile or copolymer with an acidic compound, e.g. Courtelle, Orlon, or Acrilan 16.

Fibres of the first or basic type can be dyed to relatively full colours with disperse dyes, or alternatively, they can be dyed with normal acid, metal-complex, or chrome dyes [8]. Fibres of the Courtelle type can be dyed only to pale to medium depths with disperse dyes and have no affinity for wool dyes under normal conditions, but they can be dyed to a complete range of colours with basic dyes [9].

So far as the carpet industry is concerned, selected disperse dyes have adequate fastness for almost all the colours for which they can be used, and hence it is usually not necessary to consider dyeing Acrilan or similar fibres with wool dyes if dyeing is to be carried out on yarn or piece goods. Only if dyeing is to be carried out on the loose fibre need chrome dyes be considered to give the necessary fastness to wet treatment in full colours. For the second class of fibres, e.g. Courtelle, a cationic or modified basic type of dye has to be used in medium to full depths, and here excellent fastness to light and wet treatments can be obtained with dyes of the Maxilon type.

One disadvantage which this type of dye possesses is that very little or no exhaustion occurs below 85°c., after which the dye exhausts very rapidly on to the fibre (Fig. 1). It has therefore been found necessary to use some restraining agent to minimise the effect of this critical temperature as far as possible. Cationic agents have been suggested, but their action is relatively limited. They also tend to accentuate the differences in dyeing rates between individual members of the series, and this makes dyeing with mixtures difficult, since the dyes exhaust at different stages during dyeing. We have recently put forward a new method, known as the *IT method* [10], in which a combination of anionic and non-ionic dyeing assistants is used. An anionic assistant such as Irgasol DA is the active restraining agent, and unlike the cationic assistants it minimises the difference in dyeing rates between various members

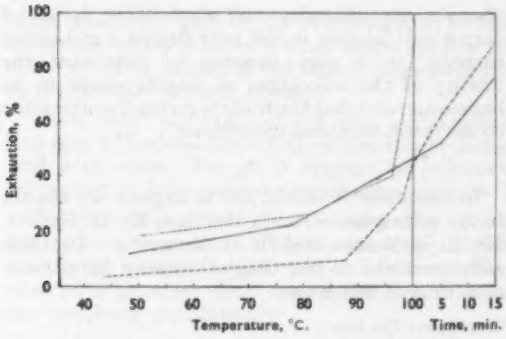

FIG. 1— Dyed in the presence of 2% Acetic Acid (40%) only

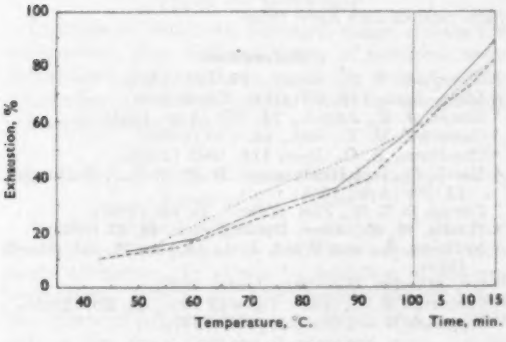

FIG. 2— Dyed by the IT Method

Maxilon Dye
——— Red BL
— — — Yellow 3GL
..... Blue GL

FIG. 1 and 2— Rates of Dyeing of Orlon with Maxilon Dyes

of the series, thus making for better compatibility in dyeing. It would be thought that it would be dangerous to use a cationic dye, i.e. a basic dye, with an anionic dyeing assistant for fear of precipitation, and this would in fact occur if the dyeing assistant were not carefully chosen. With Irgasol DA, which forms the basis of our method, this precipitation virtually does not occur except in extreme cases, but in order to prevent trouble from developing, a non-ionic dispersing agent is used such as Irgasol PMC or Tinegal VR. By means of this combination of dyeing assistants, satisfactory dyeings can be produced with the new type of basic dyes, and Fig. 2 illustrates the type of results obtainable.

Alternative Methods of Dyeing

Material for carpets can be dyed in either loose, yarn, or piece goods form. So far as conventional carpets are concerned, only the first two normally come in for consideration, but tufted carpets can be dyed in the piece[11].

Dyeing is normally carried out in the loose state only when a considerable quantity of material is required to one colour, i.e. for self colours and common ground colours. It has the advantage that batches of any size can be prepared to one colour, and uniformity of colour throughout the entire batch can be ensured by blending. Because the yarn has then to be oiled and later scoured, the dyes have to be of good wet fastness, and chrome, metal-complex, and Polar types are mainly used.

Continuous dyeing methods may be employed for loose material, and such methods have been used in practice by ourselves for both wool and Fibro[12]. A rapid uptake of dye by the fibre and quick fixation are essential, and the most suitable dyes for use on wool are the neutral-dyeing metal-complex dyes of the Irgalan type. At a pH of 3–4 good exhaustion can be obtained with these dyes in pale to medium depths in dyeing times of the order of 2–5 min. at or near the boil, and the wet fastness of these dyeings, although somewhat inferior to that of a conventional dyeing with the same dye, is nevertheless remarkable. Last year we carried out a trial in which 8,500 lb. of a loose wool blend was continuously scoured, dyed, and mothproofed in 3¼ hr., and we found it remarkably easy to control the colour of the dyeing. A five-bowl scouring machine of the Petrie McNaught type was used, the first three bowls being devoted to the conventional scouring at approx. 120°F., and the last two for dyeing and mothproofing at approx. 205°F. A drip-feed arrangement of concentrated dye solution was set up, and also further drip feeds for acid and mothproofing agent. By careful regulation of the valves, it was possible to control the pH, the colour, and also the amount of mothproofing agent taken up by the fibres.

In a trial of this nature, it is essential to have an accurate recipe prepared by the laboratory before dyeing is commenced in bulk, and we found in this case that an accurate reproduction of the laboratory recipe was obtainable. It is true that this type of dyeing is mainly limited to colours which can be obtained with metal-complex dyes and is also somewhat limited in depth by wet fastness requirements, but nevertheless a useful range of colours can be produced by this method. Only one firm in this country has, to our knowledge, carried out extensive trials with the Polar dyes, which do give a somewhat lower standard of wet fastness, particularly in full colours, and are less easy to control. Nevertheless, this method has been used repeatedly with apparently satisfactory results.

Fibro may be dyed continuously by a slightly different method, loose material being padded with the dye solution, and electrolytes being excluded from the dyebath as far as possible. After passing through the nip rollers, the material is entered into the next bath, which is a concentrated salt solution, the final bath being a rinse. This type of application is again limited to pale to medium depths, and it is of the utmost importance to choose compatible dyes for this work. Nevertheless, when carefully controlled, very good results can be obtained.

Backing of Carpets

Some carpet firms are contemplating the dyeing of the back of the carpet to a similar colour to the pile itself. We understand that this is becoming popular in the U.S.A., and it does tend to hide any cut edges.

The new types of plastic backing— polyvinyl chloride and polyvinyl acetate— are ideal for this purpose, since they can so easily be coloured to any desired shade by using a suitable pigment emulsion of the Irgalite type. Other advantages which this type of backing gives are—

(1) Firm anchorage of the pile
(2) Handle can be varied as desired
(3) Improved fire resistance
(4) Prevents attack by bacteria, etc.
(5) Does not deteriorate quickly
(6) Makes the backing waterproof
(7) Copper-free dyes need not be used.

This kind of backing is therefore of considerable interest for tufted carpets, but it has also a limited interest for the conventional woven carpets. Conventional machinery can be used for spray or roller application, or a simple device may be set up for spreading, which does allow a thicker coat to be applied. This type of backing may also have the advantage of sealing in the oil in the backing yarn and thus minimising the "wicking" which occurs on the pile yarn. If this can be achieved, it will result in a decrease in the rate of soiling of the carpet. Polyvinyl acetate can be included in the normal size to give some of these advantages, and this is proving of interest to several firms at the moment.

The carpet trade is now reacting to a spate of new ideas. There is no doubt that considerable changes are occurring and that other forms of carpet will be seen in the near future, e.g. knitted carpets. It is very pleasing to note that the quality of the coloration of carpets seems to be increasing, and that the trade is giving due attention to the latest technical discoveries.

* * *

In conclusion I should like to express my thanks to my colleagues— Mr. C. Hobday, Mr. H. Kellett, Mr. E. Bellhouse, and Mr. C. Musgrave— for their assistance and to the Geigy Company for permission to give this paper.

THE GEIGY CO. LTD.
RHODES
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(*MS. received 15th April 1958*)

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COMMUNICATIONS

Adsorption at Organic Surfaces
IV— Adsorption of Sulphonated Azo Dyes by Chitin from Aqueous Solution

C. H. GILES, A. S. A. HASSAN, and R. V. R. SUBRAMANIAN

Lobster-shell chitin has been used in quantitative adsorption studies with sulphonated azo dyes in aqueous solutions, mainly under acid conditions. The extent of adsorption has been correlated with the dye structures.

The mechanism of adsorption appears to be largely one of physical association between the aromatic residues of the dye and the molecular chains of the chitin, hydrogen bonding in acid solutions being unimportant. When inorganic acid is present, one sulphonate group in the dye anion takes part in ion exchange with inorganic anions at the cationic centres in chitin. Additional sulphonate groups appear to remain uncombined with the substrate and to reduce the affinity of the dye.

Introduction

Apparently the only previous investigations on adsorption by chitin are studies of its behaviour with organic and inorganic acids [1], protein [2], and some aromatic anionic solutes in neutral and alkaline solutions [3]. Chitin may be expected to have some of the adsorption characteristics of the cellulosic fibres and of the proteins, containing as it does both a polymer chain of substituted glucosidic residues and a high proportion of amide residues. Its reactions with solutes may therefore throw useful light on the adsorption mechanisms of both these large groups of natural polymers. Very little is known of its properties, and there is therefore a wide field of research open for exploration; the present and the two related papers can be considered a broad survey of this field. The work described in the preceding paper [1] showed that the material as normally prepared, and probably the original natural product also, may not be entirely poly-*N*-acetylglucosamine, as usually supposed, but may have about 13% of its amino groups unacetylated. It was suggested that acid anions are adsorbed at the cationic acetamido and amino groups, rather as they are adsorbed by protein fibres, and that adsorption of large aromatic anions is enhanced by the non-polar association of their planar nuclei with the molecular chains of the substrate, as it is in the adsorption of dyes by cellulose. There is a factor in chitin adsorption, however, which does not appear normally in adsorption by cellulose or protein

fibres: this is the high degree of crystallinity of the substrate, which in some circumstances appears to cause a fall in adsorption of anions proportionate to the increase in their volume.

There was no evidence of any pronounced influence of hydrogen bonding on adsorption under acid conditions [1], though it appears to influence adsorption in some neutral and alkaline solutions [3].

The present paper records a broad survey of the adsorption properties of chitin towards sulphonated azo dyes, mainly under acid conditions. The adsorption data are quantitative, and the affinities are compared qualitatively*.

Experimental

TYPES OF ISOTHERM

In order to obtain an adequate range of data for comparison, four different types of isotherm were determined (cf. methods used for wool [4]). The conclusions reached appear to be consistent amongst themselves.

(*a*) *Baths of High Constant Concentration in a Range of pH Values*

In this method saturated solutions of the free dye acids were prepared and then adjusted to a range of higher pH values by addition of sodium hydroxide. This is the best method for studying the effects of pH on adsorption, but it was used for only a few dyes because it requires more solute than was in many cases available, and because several dyes are not sufficiently soluble in acid solutions to be applied in this way. The results are shown in Fig. 1.

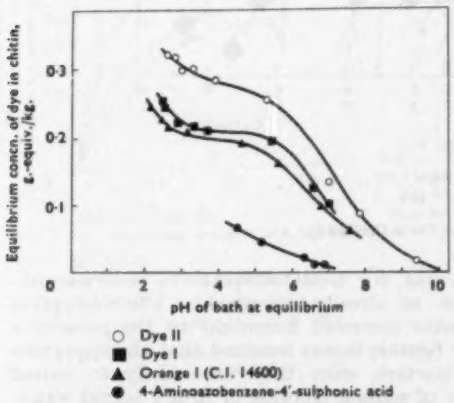

FIG. 1— Isotherms for AZO Compounds on Chitin (Method *a*) at 50°C.

(*b*) *Baths of Low Constant Concentration in a Range of pH Values*

This method, which requires smaller quantities of dye, was used with a wide range of azo dyes as a substitute for method (*a*), though the interpretation of its results is less significant.

Solutions of constant concentration (0·00011 M.) of azo compound were applied at 50° and 60°C., after adjustment to a range of pH values by addition of sulphuric acid or sodium hydroxide. This method enables a wider range of pH values to be used than does method (*a*), but the results are affected, of course, by the competition for the substrate between the acid anion and the dye anion. The curves obtained (Fig. 2 and 3; cf. Table I) are of the nature of titration curves for chitin, showing variation with pH in amount of dye bound; but because the dye concentration is kept constant and rather low, no indication of the saturation adsorption value at any pH is obtained. This value was determined by method (*c*), but at only one pH, viz. that giving maximum adsorption in method (*b*).

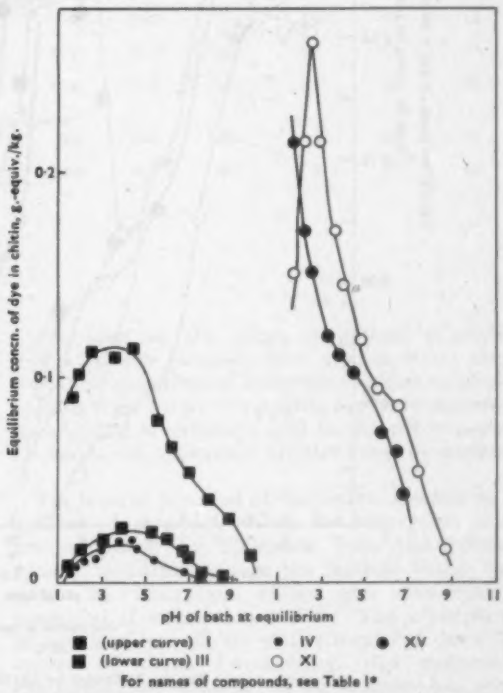

FIG. 2— Isotherms for AZO Compounds on Chitin (Method *b*) at 50°C.
*XV = Tetrasulphonated direct dye (see Experimental Section)

(*c*) *Baths of Variable Concentration and Constant pH*

A series of solutions covering a range of initial dye concentrations was used, each adjusted by addition of sulphuric acid to (or near) the initial pH value found to give maximum adsorption by method (*b*). This method is also affected, of course, by competition of the acid anion. Table I includes the maximum adsorption figures obtained by this method, determined by direct inspection of the isotherm or by a "Langmuir plot".

(*d*) *Unbuffered Baths of Variable Concentration*

This method was employed for a few dyes to determine apparent heats of adsorption (Fig. 4 and 5). No buffer was used, to avoid salting-on effects.

* The two methods hitherto used for calculating affinity data for dyes [4] are inapplicable here, because both the number of sites and the volume of available aqueous phase in the substrate vary with the volume of the anion [1].

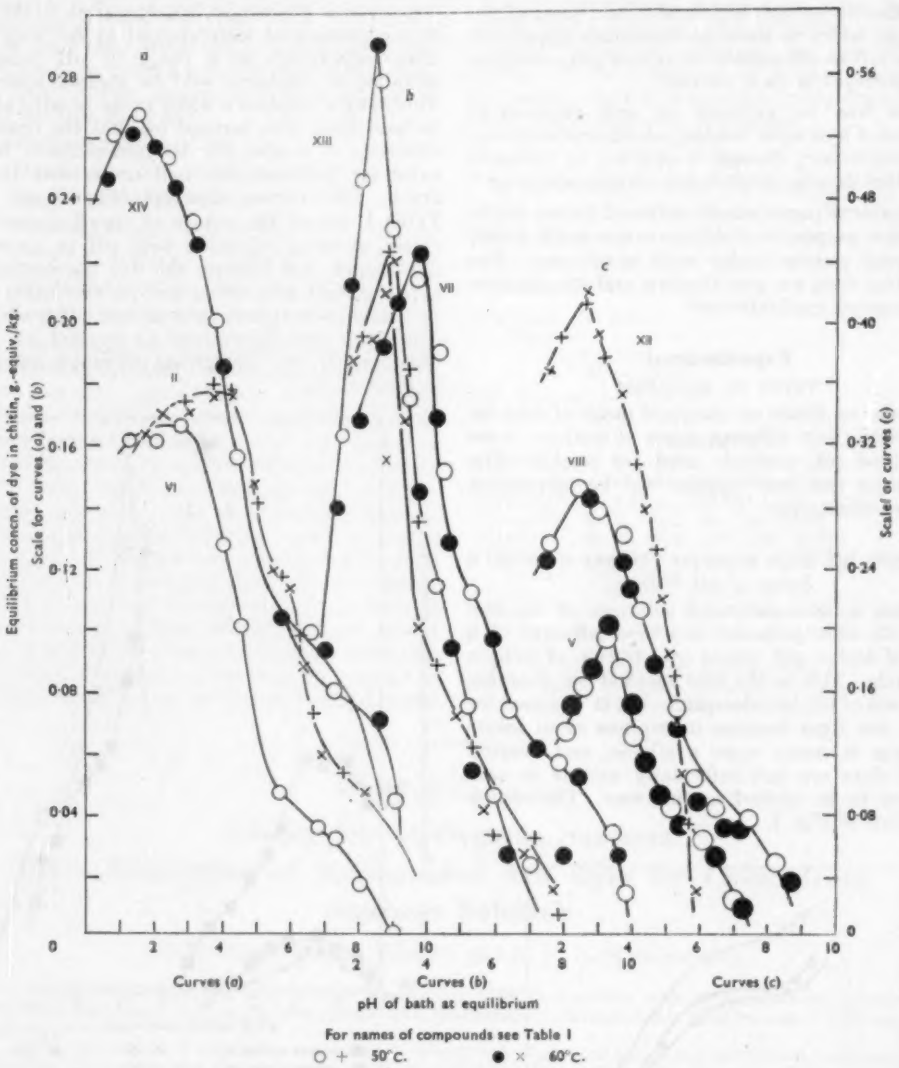

FIG. 3— Isotherms for Azo Compounds on Chitin (Method *b*)

MATERIALS AND TECHNIQUE

Chitin was prepared and used (flaked) as described in the preceding paper[1]. Some of the azo dyes were obtained in an already purified state, others were prepared by the customary methods from recrystallised intermediates, and were crystallised from water to 90–100% purity. The free acids of azo compounds for type (*a*) isotherms were obtained by precipitation of the solutions of their sodium salts with hydrochloric acid. The tetrasulphonated direct dye (Fig. 5*c*) was supplied in pure form by the manufacturers, who disclosed its constitution confidentially. Purities of the dyes were determined either by titanous chloride titration or by the acid-dichromate oxidation method[5]. The adsorption tests were made in sealed tubes, tumbled in a thermostat, for times adequate to ensure equilibration, as already described[1]. The adsorption data were corrected according to the respective purity figures; it was assumed that the impurities were inactive, since they were likely to consist largely of sodium chloride or firmly bound water. When equilibrium had been reached, the solutions were analysed, after removal of the chitin by decantation or filtration through paper. For the azo compounds tested by methods (*b*) and (*c*) 0·01 g. chitin and 20 c.c. solution were used; for those tested by method (*a*), 0·02 g. chitin and 20 c.c. solution.

Normally the amounts of solute adsorbed were determined by difference from measurements of the initial and final solution concentrations, but for the type (*a*) isotherms the measurements were

TABLE I

Adsorption of Azo Dyes by Chitin

	Dye Components: Diazo	Coupling	Colour Index No.	Approx. Mol. Vol. (A.^{3})	Purity (%)	Max. Adsorption (mg.-equiv. per kg.)*	pH†	Adsorption at pH 5·0† (m-mole per kg.)	Volume per kg. (A.^{3} × 10^{24})
			MONOSULPHONATES						
I	Sulphanilic acid	2-Naphthol	15510	700	ca. 100	165	4·0	95	41
II	1-Naphthylamine-4-sulphonic acid	2-Naphthol	15620	750	ca. 100	165	4·2	135	62
III	Azobenzenesulphonic acid		—	500	—	—	3·8	26	7·9
IV	4-Hydroxyazobenzene-4-sulphonic acid		—	550	ca. 100	120	4·2	14	4·7
			DISULPHONATES						
V	Sulphanilic acid	2-Naphthol-6-sulphonic acid	15985	850	93·5	185	3·8	40	21
VI	1-Naphthylamine-4-sulphonic acid	2-Naphthol-6-sulphonic acid	16045	950	ca. 100	330	2·5	ca. 40	ca. 23
VII	Aniline	2-Naphthol-3:6-disulphonic acid	16100	900	ca. 100	330	3·8	53	29
VIII	1-Naphthylamine	2-Naphthol-3:6-disulphonic acid	16180	900	ca. 100	305	2·6	80	43
IX	3:3'-Dianisidine	1-Naphthol-4-sulphonic acid	24140	1400	ca. 100	ca. 260‡	2·4	98	58
			TRISULPHONATES						
X	Aniline-2:5-disulphonic acid	2-Naphthol-6-sulphonic acid	—	800	91·0	315	3·0	24	12
XI	Sulphanilic acid	2-Naphthol-3:6-disulphonic acid	—	1000	91·0	245	2·6	33	20
XII	1-Naphthylamine-4-sulphonic acid	2-Naphthol-3:6-disulphonic acid	16185	1000	84·0	460(?)	2·6	50	30
			TETRASULPHONATES						
XIII	Aniline-2:5-disulphonic acid	2-Naphthol-3:6-disulphonic acid	—	950	96·5	200	2·4	21	14
XIV	2-Naphthylamine-3:6-disulphonic acid	2-Naphthol-3:6-disulphonic acid	—	1250	91·5	350	1·8	31	24

* From method (*c*) isotherms (60°C.).
† From method (*b*) isotherms (50°C.).
‡ From first inflection of isotherm.

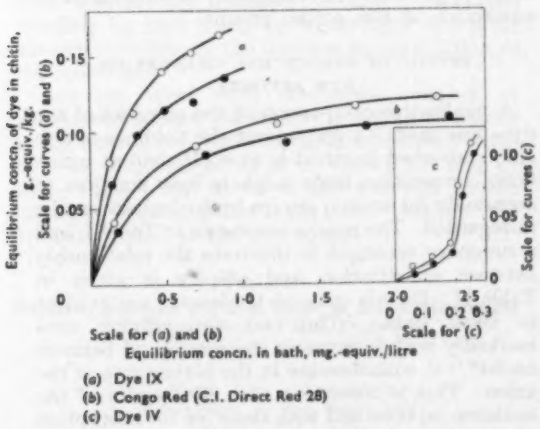

FIG. 4— Isotherms for AZO Compounds on Chitin (Method *d*)

made on mixed solutions of the substrate and dye in 80% (by vol.) sulphuric acid, as used by Rowe and Levin[6] for dissolving dyed cellulose. The dyed chitin was removed from the test solution, rinsed, dried at 105°C. for 2 hr., then placed in the acid mixture, and the whole kept in a refrigerator for 12 hr., when complete solution of the material was seen to have taken place, without charring. (The dye could not be completely extracted from the dyed chitin, even with sodium hydroxide solution followed by pyridine.)

For tests of the effect of sodium sulphate, 0·00011 M. dye solutions were used at 60°C.; they contained quantities of anhydrous sodium sulphate varying from 1·0 to 10·0 g./litre, and were adjusted by addition of sulphuric acid to the pH required for maximum adsorption as determined by method (*b*).

The level of precision of the results depends on a number of variables. When the adsorption data are calculated by difference from the optical density measurements on the residual baths, the errors in adsorption values are considerably magnified if exhaustion is low. The adsorption of solutes of low affinity can be measured down to values of about 1 m-mole/kg. No statistical examination of reliability of the methods has been made, but it is believed that errors in most of the data measured instrumentally are within ±1%.

Apparent heats of adsorption have been calculated from the data of Fig. 4 and 5 by means of the Clausius–Clapeyron equation, using two different values of the dyebath concentration corresponding to one and the same value for the concentration of dye on the chitin at the two temperatures. The apparent heat is the resultant of at least two successive heat changes, viz. those accompanying removal of dye from the aqueous phase and its attachment to the solid substrate.

APPEARANCE OF DYED CHITIN UNDER THE MICROSCOPE

Dyes do not stain the interior of the raw shell substance at all, even after treatment in acid solutions at 50°C. for 24 hr. When a microscopic

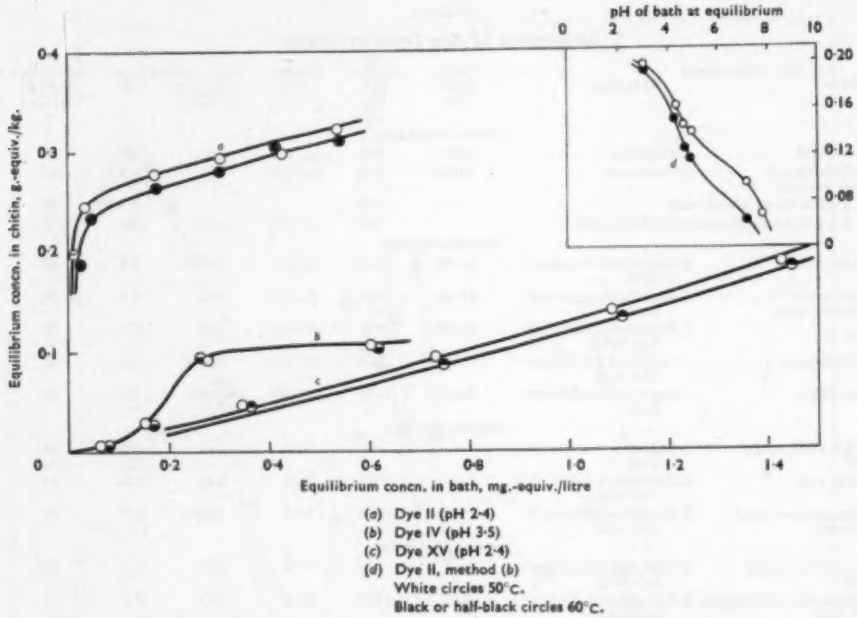

FIG. 5— Isotherms for AZO Compounds on Chitin (Method (*a*); Insert, Method (*b*))

examination is made of the raw shell after immersion in the dye solution, a thin dyed layer, presumably of protein, is seen on the inner surface, and an almost regular pattern of minute round spots containing a stainable substance, also presumably protein, is seen covering the whole outer surface. When the proteins and the mineral matter have been removed, the dye appears to penetrate the whole of the shell substance fairly evenly.

Results and Discussion

The results are shown in Fig. 1–5 and Tables I–IV.

Four dyes were examined by method (*a*). The affinity appears to rise markedly with size of the aromatic portion of the molecule (Fig. 1), which is evidence of non-polar attraction between dye and chitin. Over the range of pH values used there is no evidence that the free hydroxy group in Orange I contributes to its affinity. Indeed, this dye has somewhat lower affinity than its analogue, Orange II; perhaps the *o*-hydroxyazo chelate ring in the latter raises the affinity a little by increasing the planar area of the molecule.

All the method (*b*) isotherms (Fig. 2 and 3, Table I) have the following features— (i) A maximum somewhere in the region between pH 4·7 and 1·8, which must represent adsorption at the acetamido groups. The fall in adsorption when the acidity is increased beyond this point must be due to competition by the anion of the added sulphuric acid, or to removal of entire acetamido groups by acid hydrolysis, leaving only hydroxy groups, with a resultant fall in cationic sites, or to both causes. A similar phenomenon is observed in the adsorption of some anionic dyes on wool[4]. (ii) Besides the principal maximum, a rather poorly defined subsidiary maximum lying in the region between pH 6 and 4, which may be attributed to adsorption at free amino groups.

EFFECT OF STRUCTURAL CHANGES ON DYE AFFINITY

A qualitative comparison of the affinities of the dyes was made by setting out the volumes of dye anion adsorbed (method *b*) at a pH (5·0) at which both competition from sulphate ions and loss of acetamido (or amino) groups by hydrolysis can be disregarded. The results are shown in Table I, and a summary arranged to illustrate the relationship between constitution and affinity is given in Table II. Certain general tendencies are evident in these data. Thus (*a*) the affinity rises markedly with increase in the number of benzene nuclei*, i.e. with increase in the planar area of the anion. This is consistent with the results of the isotherm (*a*) tests and with those for the adsorption of organic acids described in the previous paper[1]. (*b*) The affinity falls with increase in the degree of sulphonation of the dye†. Thus each sulphonate group, after the first, has a negative affinity for chitin. This must mean that the second and subsequent sulphonate groups in an anion are not located at cationic sites (probably owing to steric effects), but remain dissolved in the water, thus tending to desorb the anion from the substrate. It is not surprising, perhaps, that the cationic sites are not correctly spaced to accommodate each anionic group in a polybasic anion.

* Except with dye IV, which is out of order in this sequence.

† On wool a similar effect appears to occur under some acid and neutral dyeing conditions[4,7]; and increasing sulphonation increases the ease of desorption by alkali[8], for the same reason.

TABLE II

Relation between Structure and Volume of Dye Anions adsorbed by Chitin

(pH 5·0; method (*b*); 50°C.; vol. adsorbed (A^3. × 10^{-24}) per kg.)

No. of Benzene Nuclei	No. of Sulphonate Groups			
	One	Two	Three	Four
Two	4·7, 7·9	—	—	—
Three	41	21, 29*	12, 20	14
Four	62	(23), 43*	36	24
Six	—	58	—	—

* First component unsulphonated.

TABLE III

Apparent Heats of Adsorption ($-\Delta H_{app}$) of Azo Dyes on Chitin*

Dye	C.I. No.	pH	$-\Delta H_{app}$ (kcal./mole)
4-Hydroxyazobenzene-4′-sulphonic acid	—	3·5	<0·5
4-Hydroxyazobenzene-4′-sulphonic acid	—	Neutral	1·3
Congo Red	C.I. 22120	Neutral	9·0
Chlorazol Azurine G	C.I. 24140	Neutral	9·0
Tetrasulphonated direct cotton dye	—	2·0	1·5

* Measured at an equilibrium concentration (on the substrate) of 40 m-mole/kg., temp. 50–60°C.

The introduction of a *p*-hydroxy group into azobenzenesulphonic acid raises the affinity (and the apparent heat of adsorption) (Table III). It is not clear whether the increases are due to hydrogen bonding by this group or to stronger non-polar attraction following the increase in conjugation of the molecule.

Amongst dyes with the same number of sulphonate groups, the affinity is higher when one component of the dye is unsulphonated than when both are sulphonated (cf. dyes V with VII and VI with VIII). This is no doubt a result of the stronger intermolecular attraction between the aromatic nuclei in the dye anions.

TABLE IV

Relation between pH and Value of Mean Maximum Adsorption of Azo Dyes

(Method (*b*) isotherms)

pH of Max. Adsorption	Mean Max. Adsorption (mg.-equiv./kg.)
> 4·0	155
3·0 – 4·0	275
< 3·0	330

There is a general tendency for the pH of maximum adsorption to fall with increase in degree of sulphonation or with decrease in anionic size. The lower is the pH at which maximum adsorption is reached, however, the higher is that adsorption (see Table IV). This must be a result of the increase in number of cationic sites with increase in acidity. Its effect is seen in a reverse manner in the data for monosulphonates, which, though having highest affinity, reach only low saturation values at their optimum pH, because this occurs in regions of low acidity.

COMPARISON OF ANIONIC ADSORPTIONS ON CHITIN AND WOOL

The adsorption of acids and the sulphonated dyes by chitin may be regarded as an ion-exchange process at the cationic sites. Thus, for adsorption of dye in presence of sulphuric acid the following reaction sequence may be formulated—

$$Ch{\cdot}\overset{+}{N}H_2{\cdot}CO{\cdot}CH_3)_2SO_4^{--} + NaDy \longrightarrow Ch{\cdot}\overset{+}{N}H_2{\cdot}CO{\cdot}CH_3)Dy^- + \tfrac{1}{2}Na_2SO_4$$

(Ch = chitin residue; Dy = dye anion).

Little appears to be known regarding heat changes in ion-exchange adsorption mechanisms with organic solutes, but in some types of ion-exchange adsorption for which data are available, viz. sulphate esters on γ-alumina [9], basic dyes on silica [10] and on graphite [11], and some adsorptions on resins (quoted in reference [9]), there is a very low apparent heat of adsorption. Results here show (Fig. 2 and 3, Table II) that monoazo dyes have very low apparent heats of adsorption, i.e. their isotherms show almost no change with change in temperature, which is thus consistent with ion exchange*. Ion-exchange adsorptions in which one of the adsorbed ions is held to the surface by other strong associative forces may, however, exhibit appreciable apparent heat changes. Thus in the present tests disulphonated dyes with large anions have considerable apparent heats of adsorption (Table III). The cause may be forces of association between the anion and the chitin molecular chain, or between the dye anions themselves at the interface.

Somewhat similar phenomena are observed with wool. Thus Derbyshire and Peters [12] have found, by direct calorimetric measurement, that the first adsorption of hydrochloric acid (40 m-mole/kg.) from water by wool at 25°C. occurs with negligible evolution of heat. (They ascribe the reaction to back titration of free carboxylate ion sites, which are present in small amount accompanied by a corresponding quantity of free cationic histidine groups, with which the chloride ion will then become associated, presumably also with negligible heat evolution.) With increase in uptake of acid beyond this amount the heat change is constant at about −2·8 kcal./mole of acid bound, attributed to breakdown of amino–carboxylic acid salt links in the fibre by back titration of the carboxylate groups, following adsorption of hydrogen ions. Apparent heats of adsorption of the anions of dyes and organic acids on wool are also quite appreciably larger than the value for hydrochloric acid, figures between −3 and −11 kcal./mole being given in the few recorded cases [4, 13–16]†.

The apparent heats of adsorption on chitin seem to fall with an increase in acidity of the solution (Table III, Fig. 5). Hackman [3] found that the apparent heat of adsorption of water-soluble

* The heat of ionisation of the acetamido or amino group would be of the order of −5 kcal./mole, but this does not, of course, influence the present results, because the amount of dye anion, not the amount of acid cation, adsorbed is the quantity measured.

† Derbyshire and Marshall [16] suggest that for Orange II the high value may be the result of the large heat of dilution of the dye.

protein on chitin is very low, i.e. temperature change has little effect on the adsorption, which is nil on the alkaline side of pH 9 and rises to a maximum at about pH 5.

ADSORPTION OF AZO DYES IN PRESENCE OF SODIUM SULPHATE

A number of experiments were made in which chitin was treated with an azo dye (as the sodium salt) in presence of sulphuric acid and sodium sulphate in various concentrations. Under these conditions when dyes are applied to wool, there is a linear relation between $\log C_f/C_s$ and $\log C_{SO_4}$, where C_f and C_s are the equilibrium concentrations of dye anions in the wool and in the solution, and C_{SO_4} is that of sulphate ions in the solution, the slope being equal to $z/2$, where z is the valency of the dye anion [7]. The present results also gave linear plots of this type, but the slopes generally had lower values than the theoretical. There was some vertical displacement of the plots, indicative of differing anion affinities roughly corresponding with the expected order.

CATIONIC SOLUTE

An adsorption isotherm determined with a cationic compound (cetyltrimethylammonium bromide) showed that the amount of anionic sites is insignificant, being of the order of 20 mg.-equiv./kg.

* * *

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The Dyeing of Cellulose Acetate with Disperse Dyes
VII—A Comparison with their Solubility in Organic Solvents

C. L. BIRD

The well known parallel between the solubility of disperse dyes in certain organic solvents and their suitability for dyeing secondary cellulose acetate has been examined. It is shown that, at room temperature, a close relationship is obtained only by using a solvent containing an appreciable amount of water, e.g. 20% aqueous diethylene glycol diacetate (DEGDA).

INTRODUCTION

According to Mellor and Olpin [1], "solubility in esters such as ethyl acetate is a reliable guide to the probable behaviour of a dye towards acetate rayon".

Earlier, Kartaschoff and Farine [2] found an analogy, at 60°C., between solubility in ethanol and solubility in secondary cellulose acetate. They used 15 anthraquinonoid disperse dyes and related compounds, but the method employed for determining solubility in the fibre was unsatisfactory. Dyeings were carried out for only 2 hr., and an unstabilised suspension was used, with the result that there was much superficially adsorbed dye, which had to be removed by soaping at 40°C. Statistical analysis of their results, leaving out of account alizarin, purpurin, and two compounds which were not appreciably adsorbed by the fibre, shows that the correlation is not significant.

Recently, Bird and Miss Harris [3] have given a number of fibre saturation values for disperse dyes on secondary cellulose acetate at 80°C.* For some of these dyes heats of solution in the fibre were also given, thus enabling the solubility at room temperature to be calculated. These calculated values can be compared with the solubilities of the dyes in organic solvents at the same temperature.

If there is an exact parallel between cellulose acetate and ethyl acetate, a plot of the solubilities

* Partovi [4] has obtained equilibrium isotherms up to saturation at 60° and 80°C. for a number of disperse dyes. His results indicate that Bird and Harris's calculated saturation values are substantially correct.

TABLE I

Solubilities (g./litre)

No.	Dye	Secondary Cellulose Acetate (calc.)	Ethanol	Ethyl Acetate	Benzene	20% Aqueous Ethyl Acetate–Ethanol	20% Aqueous DEGDA
	SECTION A						
I	4:4′-Diaminoazobenzene	20·0	4·7	19·5	0·65	21·2	23·1
II	4-Nitro-4′-aminoazobenzene	5·1	1·8	14·7	1·6	6·0	7·9
III	3-Nitro-4′-aminoazobenzene	4·1	0·62	4·5	0·40	1·3	4·6
IV	4-Nitroaniline→ *N*-ethyl-*N*-β-hydroxyethylaniline	2·6	2·8	9·6	2·2	9·3	7·3
V	4-Nitroaniline→*NN*-bis-β-hydroxyethylaniline	2·2	0·58	0·68	0·05	1·6	1·9
VI	2-Methoxy-4-nitroaniline→ *NN*-bis-β-hydroxyethylaniline	39·5	2·8	3·5	0·32	24·6	19·8
VII	2:6-Dichloro-4-nitroaniline→ *NN*-bis-β-hydroxyethyl-*m*-toluidine	6·0	9·9	11·5	1·1	33·2	18·4
VIII	2:4-Dinitro-4′-hydroxydiphenylamine	35·5	13·4	54·9	0·73	58·7	81·5
IX	1:4-Diaminoanthraquinone	3·5	1·2	3·6	0·39	1·0	1·3
X	1:4-Diamino-2-methoxyanthraquinone	8·5	0·68	1·8	0·35	2·9	5·3
XI	1-Amino-4-hydroxyanthraquinone	1·7	0·83	5·7	1·8	1·9	3·6
XII	1-β-Hydroxyethylaminoanthraquinone	16·5	2·6	4·6	0·78	8·0	7·6
XIII	1:4-Bis-β-hydroxyethylaminoanthraquinone	1·3	0·18	0·08	0·003	1·5	1·5
	Slope (log–log)	—	0·75	0·82	0·46	0·95	0·86
	*Correlation coefficient (log–log)	—	0·70	0·57	0·31	0·77	0·82
	SECTION B						
XIV	4-Nitroaniline→diethylaniline	ca. 1·0	0·62	9·4	25·2	1·4	—
XV	4-Nitroaniline→diphenylamine	ca. 10	4·3	52·3	16·9	7·8	—
XVI	1-Methylaminoanthraquinone	2·6	0·58	5·6	9·8	1·7	—
XVII	1:4-Bismethylaminoanthraquinone	1·1	0·12	0·86	1·9	0·58	—
XVIII	1-Methylamino-4-anilinoanthraquinone	ca. 1·5	0·49	8·1	26·3	1·6	—

* For eleven degrees of freedom, any correlation coefficient exceeding 0·553 is significant at 5% level of significance.

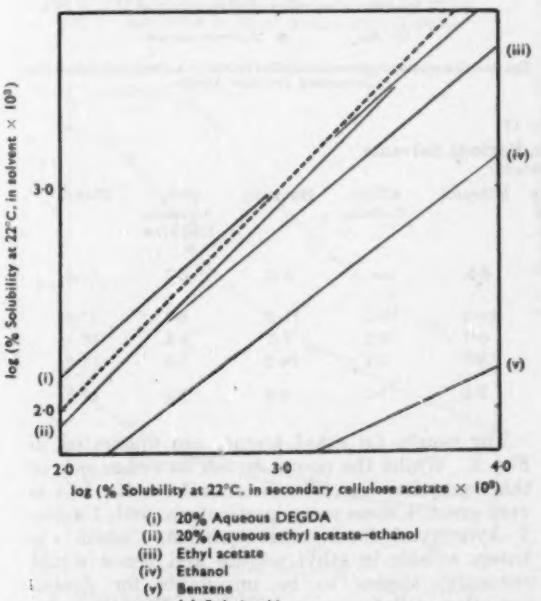


FIG. 1—Best Straight Lines

in the two solvents will give a straight line of unit slope passing through the origin. In order to discover whether such a relationship exists, solubilities of a range of disperse dyes have been determined at 22°C. in ethyl acetate, as well as in other organic solvents. The results are given in Section A of Table I, in which the calculated values for secondary cellulose acetate are based on the air-dry weight (6·6% moisture), using a density value of 1·31 g./c.c. In Section B are given figures for some of the more hydrophobic disperse dyes; in these cases heats of solution in the fibre are not known, and the figures for solubility in the fibre at 22°C. (with two exceptions, derived from Partovi's results[4]) are only rough estimates based on an average value for the heat of dyeing.

For calculating the correlation coefficients (Section A) a log–log transformation has been used, since it is the *ratios* between the solubilities

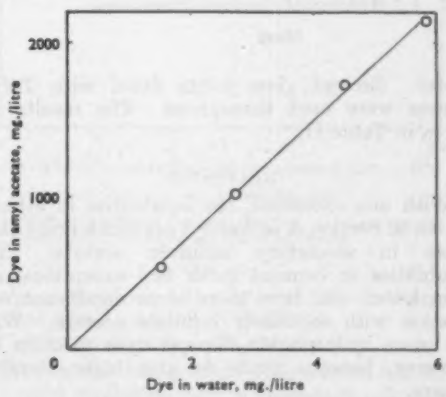


FIG. 2—Partition of 2-Methoxy-4-nitroaniline→*NN*-bis-β-hydroxyethylaniline between Amyl Acetate and Water at 22°C.

which are of interest. The slopes of the best straight lines through the points are given in Table I and are illustrated in Fig. 1.

An additional experiment consisted in determining the partition coefficient of 2-methoxy-4-nitroaniline→*NN*-bis-β-hydroxyethylaniline between water and amyl acetate at 22°C. A straight-line relation up to saturation in both phases was obtained, as shown in Fig. 2. Solid dye did not appear at the interface until the solutions had reached saturation.

EXPERIMENTAL

Purified disperse dyes, prepared as described in Part I [5], were left in contact with the solvent in glass-stoppered test tubes for 4 weeks in a room maintained at 22°C. The tubes were shaken once every day. A 2-ml. sample of each solution was then removed after settling for 24 hr., by means of a pipette fitted with a small cotton wool plug to remove any solid dye. The sample was then diluted with acetone, and the dye present was estimated colorimetrically by means of a Hilger Spekker absorptiometer.

The solvents used were "absolute" alcohol, and AnalaR ethyl acetate, amyl acetate, and benzene. The sample of diethylene glycol diacetate (DEGDA) used was kindly supplied by the late Mr. H. C. Olpin and had been decolorised with activated charcoal*.

Heats of solution in various solvents were determined for four disperse dyes from solubilities at 30°, 40°, and 50°C. The apparatus consisted of a thermostated 50-ml. flask fitted with a side arm, a condenser terminating in a silica gel tube, and a stirrer driven through a mercury seal by an electric motor. Ground glass joints fitted with Teflon sleeves were used throughout. The results are given in Table II.

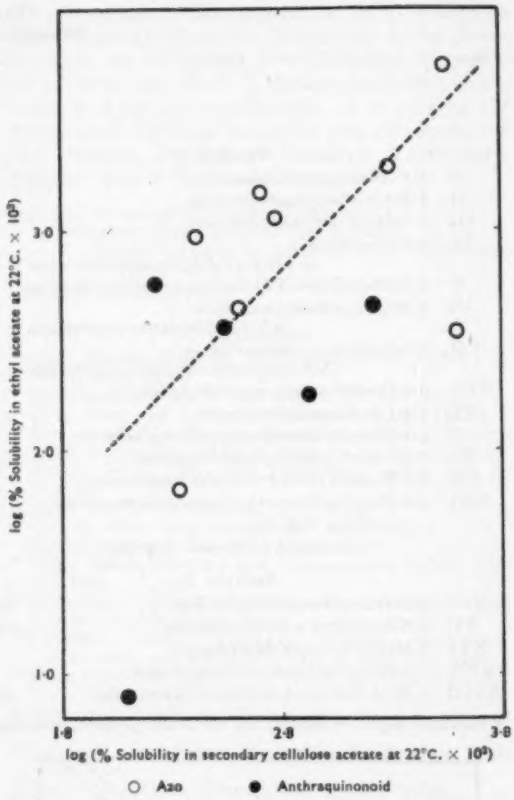


FIG. 3—Correlation between Solubility in Ethyl Acetate and Solubility in Secondary Cellulose Acetate

TABLE II

Heats of Solution in Various Solvents

(kcal./mole)

	Dye	Secondary Cellulose Acetate (calc.) [3]	Ethanol	Ethyl Acetate	Benzene	20% Aqueous DEGDA	Water [3]
II	4-Nitro-4′-aminoazobenzene	4·8	6·3	—	8·5	6·7	15·4
V	4-Nitroaniline→*NN*-bis-β-hydroxyethylaniline	7·3	10·2	10·3	11·2	6·1	17·6
IX	1:4-Diaminoanthraquinone	5·1	6·0	5·2	7·6	9·5	15·5
XII	1-β-Hydroxyethylaminoanthraquinone	2·2	9·5	7·6	10·2	7·5	13·5
	Mean	4·9	8·0	7·7	9·4	7·5	15·5

DISCUSSION

With one exception, the solubilities in ethanol shown in Section A of Table I are much lower than those in secondary cellulose acetate. The solubilities in benzene (with two exceptions) are even lower, and here there is no significant correlation with secondary cellulose acetate. With the more hydrophobic disperse dyes (Section B), however, benzene tends to give high solubility figures.

The results for ethyl acetate are illustrated in Fig. 3. Whilst the points do fall on either side of the theoretical line, the distance from the line is very great in some cases, particularly with 1:4-bis-β-hydroxyethylaminoanthraquinone, which is barely soluble in ethyl acetate and hence would (wrongly) appear to be unsuitable for dyeing secondary cellulose acetate rayon. With the more hydrophobic disperse dyes (Section B), ethyl acetate gives values which are too high, so it may be concluded that solubility in ethyl acetate is not a very reliable guide.

Secondary cellulose acetate, however, contains both acetate and hydroxyl groups, so for comparison a mixture of ethyl acetate and ethyl alcohol is required. Further, in a dyebath, the acetate and

* DEGDA is manufactured by British Celanese Ltd.

hydroxyl groups will be hydrated. A mixture of ethyl acetate and ethanol in which the acetate : hydroxyl ratio is the same as in secondary cellulose acetate dissolves only about 10% water, which is insufficient to enable the effect of adding water to the solvent mixture to be studied over a considerable range. A mixture of ethyl acetate (100 ml.) and ethanol (60 ml.) was therefore used. This mixture contains nearly equivalent amounts of acetate and hydroxyl groups. It is a better solvent than ethyl acetate for the more hydrophilic disperse dyes, and a worse one for the more hydrophobic dyes.

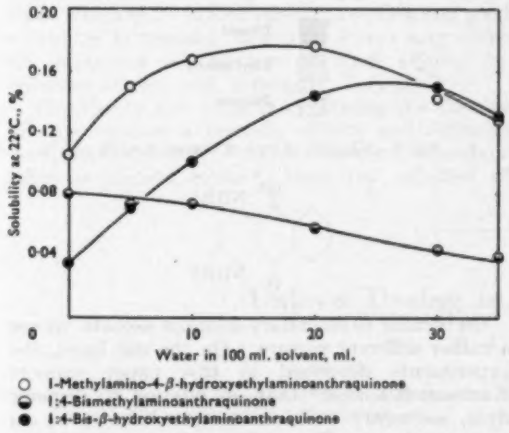


FIG. 4—Solubilities in Ethyl Acetate–Ethanol–Water

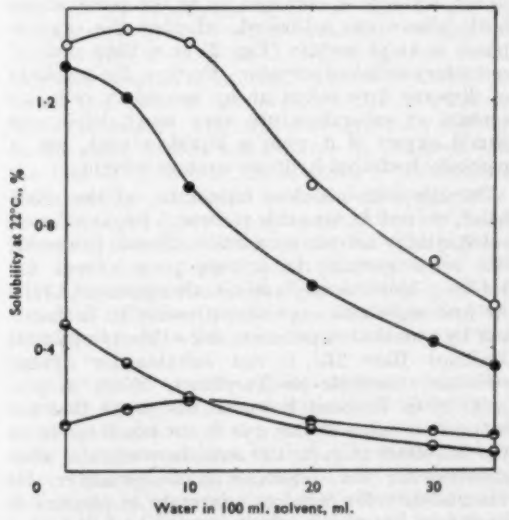


FIG. 5—Solubilities in Ethyl Acetate–Ethanol–Water

Fig. 4 and 5 illustrate the effect on the solubility of seven disperse dyes of increasing amounts of water in the solvent mixture. It is seen that the presence of water increases the solubility when the dye contains a β-hydroxyethyl group. The effect is particularly marked when the dye contains two of these hydrophilic groups. On the other hand, with dyes which do not contain β-hydroxyethyl groups, addition of water to the ethyl acetate–ethanol mixture decreases the solubility, to an extent which is much greater than can be accounted for by dilution of the mixture with a non-solvent.

Solubilities were next determined for a number of disperse dyes in a mixture prepared by diluting 20 ml. of water to 100 ml. with a mixture of ethyl acetate (100 ml.) and ethanol (60 ml.). The amount of water was chosen after trial and error, and is slightly more than equivalent to the acetate and hydroxyl groups present. As seen in Table I (Section A), the correlation with solubility in secondary cellulose acetate is better than that for ethyl acetate, with three exceptions (III, VII, and IX). The data in Section B also indicate a better correlation.

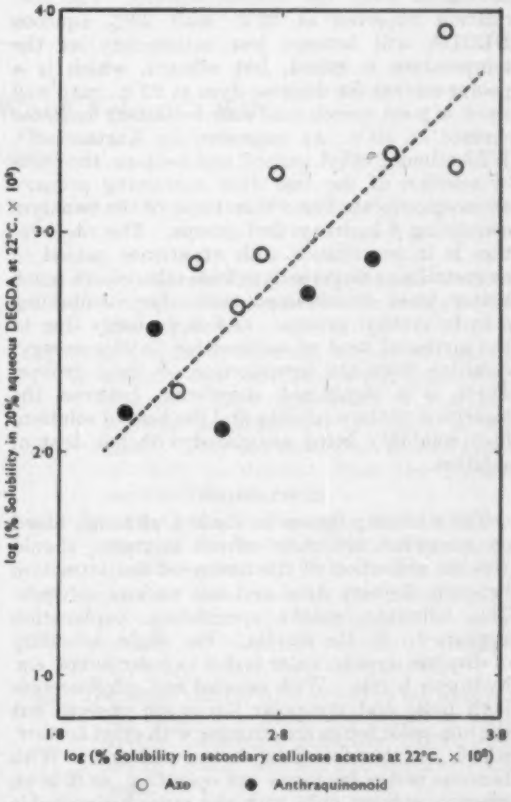


FIG. 6—Correlation between Solubility in 20% Aqueous DEGDA and Solubility in Secondary Cellulose Acetate

At the suggestion of the late Mr. H. C. Olpin, diethylene glycol diacetate (DEGDA)—

$$CH_3{\cdot}CO{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}CH_2{\cdot}O{\cdot}CO{\cdot}CH_3$$

was examined for suitability. This solvent has an acetyl value similar to that of secondary cellulose acetate. It contains no hydroxyl groups, but the ether oxygen atom will be hydrophilic. Mixtures

of water and DEGDA gave solubility curves for IV and V of the same type as those obtained with aqueous ethyl acetate–ethanol (Fig. 5). A series of solubility determinations were carried out with 20% (by vol.) aqueous DEGDA, and the results are given in Table I, illustrated by Fig. 6. The correlation is significant and somewhat better than that obtained with the 20% aqueous ethyl acetate–ethanol mixture, but the slope of the best straight line through the points (Fig. 1) is not quite as satisfactory. However, in view of the considerable amount of extrapolation involved in calculating the solubilities in secondary cellulose acetate at 22°C., the two solvent mixtures may be considered equally satisfactory.

The heats of solution in Table II are insufficient to permit any firm conclusions to be drawn, but the mean values indicate that secondary cellulose acetate has an appreciably lower average heat of solution than the other solvents, which is to be expected in view of the lower entropy change associated with the fibrous solvent. The correlation observed at 22°C. with 20% aqueous DEGDA will become less satisfactory as the temperature is raised, but ethanol, which is a poorer solvent for disperse dyes at 22°C., may well show a good correlation with secondary cellulose acetate at 60°C., as suggested by Kartaschoff[2]. With ethanol, ethyl acetate, and benzene, the heats of solution of the two dyes containing primary amino groups are lower than those of the two dyes containing β-hydroxyethyl groups. This observation is in accordance with experience gained in recrystallising disperse dyes from ethanol— a much better yield is obtained with dyes containing β-hydroxyethyl groups— and is probably due to the increased heat of sublimation (lattice energy) resulting from the introduction of these groups. There is a significant correlation between the logarithm of the solubility and the heat of solution, high solubility being associated with low heat of solution.

CONCLUSIONS

The solubility figures in Table I, although based on somewhat arbitrary solvent mixtures, should give an indication of the nature of the attraction between disperse dyes and the various solvents. The following, rather speculative, explanation appears to fit the results. The slight solubility of disperse dyes in water is due to polar forces, e.g. hydrogen bonds. With ethanol and ethyl acetate both polar and non-polar forces are present, but the non-polar forces are stronger with ethyl acetate, which is therefore the better solvent. With benzene non-polar forces are operating, so it is an effective solvent only with the more hydrophobic disperse dyes, e.g. XIV. With certain dyes, e.g. V and XIII, solution appears to depend largely on polar forces, and the presence of an appreciable amount of water is necessary in order to increase these forces and so promote greater solubility; benzene is ineffective.

This concept is illustrated in Fig. 7 with anthraquinonoid dyes containing 0, 1, and 2 β-hydroxyethyl groups. A similar chart is given in the azo series by XIV, IV, and V.

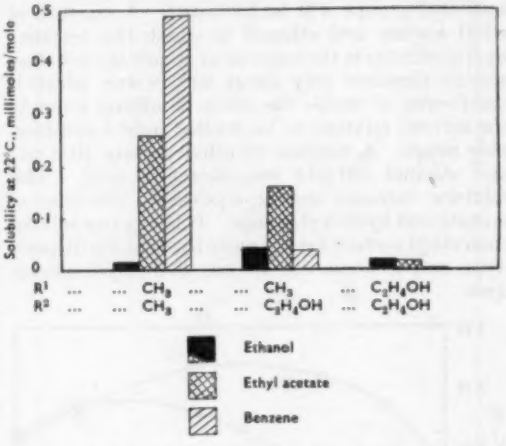
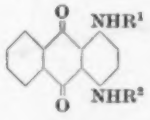


FIG. 7— Solubility of Dyes of General Formula—



On turning to secondary cellulose acetate, we see a rather different picture. On the one hand, the experiments described in this paper support Kartaschoff's view[6] that, in relation to disperse dyes, secondary cellulose acetate behaves as an organic solvent. Thus, when a disperse dye is distributed between water and an organic phase, a linear isotherm is obtained up to the point where both phases are saturated, whether the organic phase is amyl acetate (Fig. 2) or a fibre such as secondary cellulose acetate. Further, the amounts of disperse dyes taken up by secondary cellulose acetate at saturation are very much what one would expect if it were a liquid solvent, viz. a partially hydrated hydroxy–acetate solvent.

Results with cellulose triacetate, on the other hand, do not fit into this picture. Regarded as a substantially anhydrous acetate solvent, it should, like ethyl acetate, be a very poor solvent for 1:4-bis-β-hydroxyethylaminoanthraquinone(XIII). At first sight this expectation seems to be borne out by practical experience, since this dye (Serisol Brilliant Blue 2B) is not suitable for dyeing cellulose triacetate (or Terylene). Work in progress by G. Tabbron, however, has shown that the saturation value of this dye is not much lower on the triacetate than on the secondary acetate, after allowing for the difference in accessibility. Its unsuitability for cellulose triacetate in practice is explained by its low affinity at the higher dyeing temperature used with this fibre, and its very low rate of dyeing on the triacetate, which together result in a poor "building up" property.

It seems clear that cellulose triacetate, like secondary cellulose acetate, is a much better solvent than ethyl acetate for the more hydrophilic disperse dyes. This difference is most readily explained by assuming that the hydrogen bonding power of the acetyl group is greater in cellulose

acetate than in ethyl acetate and that, as suggested by Majury [7], the origin of the affinity is in the acetyl rather than in the hydroxyl group. An alternative explanation is the existence of non-polar, van der Waals adsorption forces between the dye and the fibre, as suggested by Derbyshire and Peters [8]. Such forces would not operate to anything like the same extent between disperse dyes and the small molecules of the liquid solvents. The first explanation appears more likely. The low vapour pressure of the more hydrophilic disperse dyes and their low solubility in non-polar solvents indicate internal polar bonding. On the other hand, with the more hydrophobic disperse dyes, which show higher vapour pressures and good solubility in benzene, non-polar forces may make an important contribution to their affinity for cellulose acetate and, especially, Terylene [8].

This theory also helps in explaining the absence of any correlation between affinity and diffusion coefficient in the case of disperse dyes and secondary cellulose acetate rayon [9], since the affinities of individual dyes can be compared only if the type of bonding is the same.

* * *

The dyes used in this work were kindly supplied by British Celanese Ltd., Imperial Chemical Industries Ltd., Dyestuffs Division, and The Yorkshire Dyeware & Chemical Co. Ltd.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING
THE UNIVERSITY
LEEDS 2

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Unlevel Dyeing in Wool Velour Cloth

F. J. PARKER

It has been found that mechanical abrasion can be a cause of unlevelness in the dyeing of woollen cloth with anthraquinonoid acid dyes.

For many years woollen piece dyers have been haunted by spasmodic spells of unlevel pieces, particularly in the velour qualities ranging in weights from 13 oz. to 21 oz., which required a considerable amount of milling. The unlevelness has taken the form of warpwise streaks showing up lighter or darker. The streaks are generally slightly on the slant, and do not coincide on the back and the face of the cloth. Such spells of unlevelness have usually terminated with such verbal conclusions from the dyer as "having cured itself with the change in blend" or "running out with the blend", but there have been no definite reasons for the start or, indeed, the finish of the trouble. With a thankful sigh the trouble has run its course, but the dyer has been left with the fear of its starting again.

This type of trouble appears to be mainly confined to dyeings obtained with level-dyeing acid blues and violets of the anthraquinone type or with a large proportion of such a blue or violet in the recipe, e.g. sky blues, royal blues, mushrooms, coffees, greys, and browns. Notwithstanding the use of the most level-dyeing blues and violets of this class, such unlevelness will persist despite the wiles and the resourcefulness of the dyer. At times he may feel that he is obtaining improved results from a particular modification of technique, in either dyeing, scouring, etc., but more often than not his hopes are dashed after the next set of trials.

It would seem useful to relate these spells of unlevelness to certain facts—

(1) They coincide with periods when the various "royal blues" are in fashion and hence in great demand.

(2) The trouble is confined mainly to heavily milled velour qualities, which quite often contain a high proportion of noil and possibly a proportion of waste in the blend. They are the softer, merino types of cloth as distinct from the harder, crossbred types.

(3) At other periods of the year, when different types of shades are in vogue, these same qualities give very little trouble at all.

(4) Analytical tests made on the unlevel cloth indicate that the preparation is satisfactory, no abnormal amount of residual oil, soap, or lime soap being found.

(5) The same difficulty is experienced whether the cloths are carbonised before or after dyeing, although generally carbonising before dyeing does seem to aggravate the fault.

The following is an account of an investigation into the possible causes of this type of fault which the author has made at the works at Mytholmroyd, in collaboration with the Dyehouse Department of Imperial Chemical Industries Ltd., Dyestuffs Division, at Blackley, Manchester 9. It is hoped that it will be of some assistance to other processers of wool velour cloth.

Pieces which had been scoured normally, soap-milled and washed-off, dyed, and then carbonised

after dyeing gave streaky results. A new milling soap was tried without improvement. Subsequently, grease milling and acid milling were tried, again with no improvement. A trial lot of pieces was sent off to a commission finisher for scouring and milling, but these again gave streaky results when dyed at Mytholmroyd. Similarly, a trial lot of pieces prepared at Mytholmroyd was sent out for commission dyeing, and these also were found to dye streaky, as were pieces sent out for the complete commission preparing and dyeing operations. The presence of lime soaps, metals, etc. was carefully checked, and the use of lime-soap-dispersing agents and metal-sequestering agents tried during scouring, washing off, and dyeing, but with no improvement. A careful check on pH values of dyebaths was carried out using varying amounts of sulphuric, formic, phosphoric, and acetic acids, but with no significant difference in the results obtained. Dyeings were also made using a different water supply, but both this and the normal mill supply gave a satisfactory analysis, and the streaky results were obtained with both.

Detergent scouring was carried out, also solvent scouring, using the trichloroethylene technique at the ICI General Chemicals Division plant at Runcorn. These latter pieces were subsequently milled and washed off on the dolly, but again the result obtained on dyeing was not completely satisfactory.

Carbonising before and after dyeing, together with carbonising immediately after scouring and before milling and dyeing, all gave imperfect results. The full range of level-dyeing acid blues was tried. Obviously, in order to meet the requirements of light fastness, these were all of the anthraquinone type, and, since many of the royal blues demanded were very bright, often only the brightest members of the range, such as Lissamine Ultra Sky R and Solway Ultra Blue B (C.I. Acid Blues 67 and 25), could be used. Even with the addition of various level-dyeing assistants, streaky results were still obtained with all the blues, including the most level-dyeing types.

Unlevel pieces would not "cobble" with a normal sulphuric acid levelling boil. However, earlier suspicions that the trouble might have been arising from the presence of calcium or lime soap in the cloth had led to our trying a boiling treatment with weak hydrochloric acid (3 pints per 70-lb. piece). This rather desperate remedy was found to correct some 80% of the rejects, although it was subsequently proved by analytical tests that calcium was not present in any significant amount. At this stage it may be appropriate to observe that the vitriol-dyeing blues, applied in the presence of phosphoric, formic, or particularly acetic acid, whilst giving slower strike and exhaustion, did not subsequently respond to the hydrochloric acid cobbling treatment. This observation will be referred to later.

In order to determine whether or not the milling operation was a factor concerned in the production of the unlevel results, pieces were dyed immediately after scouring and before milling. We were surprised to note that these pieces in their open, flimsy, unmilled state, which made penetration much easier, showed the familiar unlevelness exactly to the pattern of the normal milled dyed pieces: there were the same warpwise streaks of light and dark areas, and in this case the edges of these streaks seemed to be straighter and more clearly defined. In addition there was the suspicion that the dark streak appeared to have more cover or nap than the light streak. Microscopical examination, however, revealed the presence of badly damaged fibres in both light and dark areas, and it could not be established by this means whether there was any greater preponderance of damaged fibres in the dark areas than in the light areas. The Kiton Red G test also was inconclusive.

This experiment, viz. dyeing before milling, was repeated, and the same result obtained. The apparent extra cover or surface nap on the dark streaks was again in evidence, and, with a view to protecting the surface of the cloth, a further trial lot of pieces was processed, but this time they were bagged (i.e. sewn selvedge to selvedge with the face inside) right at the start of processing, i.e. in the grease. Immediately a very different result was obtained, the back side or exposed side of the cloth again showing the typical unlevel streaks, while the protected face showed a very marked improvement, with only two streaky places where obviously the cloth had been subject to considerable abrasion or chafing.

This opened up an entirely new approach to the problem. All the dolly scouring machines in use at the mill are of the normal type such as have been made by various manufacturers over the past 25–30 years. The top roller is not positively driven, but runs only by virtue of friction drive from the bottom roller or, to be more accurate, by friction drive from the cloth as it goes through the rollers. The questions now to be answered were whether slippage or skidding of this top roller could be causing friction or abrasion unevenly throughout the cloth while in rope form, and whether this damage was the cause of the unlevel dyed appearance. A driven top roller was then tried in the scouring of a set of pieces, the pieces all being bagged right at the start, i.e. before processing in rope form. Some of the pieces were dyed (still in the bagged state) immediately after scouring, while others went on for milling (still bagged) before dyeing. All the pieces on examination after dyeing showed a practically perfect result on the protected face side, but the typical streaky unlevelness was still apparent on the back of the cloth, which was not protected by the bagging.

In view of this result, and recalling the care, time, and trouble taken in the past with the scouring and washing-off of the pieces, it was now obvious that more time spent on these processes would only tend to increase further the risk of abrasion and uneven damage. Further trials were then made cutting the time taken in these processes still using a driven-top-roller dolly and slowing down the speed of the cloth travel very considerably from the normal running speed of around 100 yd./min. Each successive test showed a

pronounced improvement, and although the back side of the cloth still tended to show a little streakiness, the face side (protected by bagging) improved until it was absolutely perfect.

The true nature of the fault was now realised. It would appear that many, if not a large majority of, velour and woollen blends are susceptible to this abrasion damage, and this damage (whether entirely physical or partly chemical is not yet known) is particularly liable to be shown up when dyeing with anthraquinone acid blues. Thus a combination of demand for royal blue fashion shades with the production of a relatively short-fibre blend cloth can give rise to the worst periods of unlevel dyeing faults. Dyes which are not susceptible to this type of damage are triphenylmethane greens and blues (which always give perfect results) and the metal-complex types applied with a high percentage of sulphuric acid. The level-dyeing acid yellows, oranges, and reds (mainly azo dyes) do not show uneven dyeing on this type of damage, and the effect seems to be confined to the anthraquinone acid blues and violets. In this latter class even the monosulphonated members, which normally give good cover of uneven chemical damage on wool, do not seem to have any advantage in covering this physical abrasion damage.

The relative success of the hydrochloric acid cobbling method would appear to depend upon the fact that this drastic treatment is adding to the damage already present, and in this way tending to level up the total damage, thus minimising the original differences. This could account for the lack of success of this treatment following upon dyeing processes using the weaker acids, which did not produce the same degree of initial overall damage as sulphuric acid.

Experiments are still proceeding in an endeavour to find suitable mechanical processing conditions for achieving a satisfactory scour without mechanical abrasion damage. The ideal would be to attain conditions such that both faces of the cloth will be satisfactory without the necessity for bagging, but in some cloths this will not be possible without modification of the blend. Incidentally, we have established that the actual milling process does not add to the damage already caused in dolly scouring, probably because the weights and pressures involved in milling are not so great, and, since the top roller in the milling machine is driven, the risk of slippage is less.

The dyer of woollen piece-goods can now take stock of his experiences over many years. At one time or other he has blamed the blends, the scouring, the washing-off, the milling, and many other factors, knowing that everything possible was being done in his dyehouse, and never really quite knowing at what stage in processing the "house had to be put in order". The present findings explain why he could process an old stock piece along with a new blend, and get perfect results from the old piece and yet have the new blend unlevel. They also explain why, to use an old scourer's phrase, a "mucky" scour sometimes gives level results: such a scour would generally cut down the processing time and, therefore, reduce the risk of abrasion damage. These findings also explain why in the past the trouble has disappeared with the change in blend or, perhaps unobtrusively, with the change in trend of colour fashion. These experiments on the mechanical conditions in scouring have drastically re-oriented the writer's thoughts and ideas on preparation techniques, and he feels that too much emphasis has been placed in the past on chemical factors and not enough on physical and mechanical factors. How many of us who keep a careful watch on our own preparatory processes have had unlevel results and can truthfully say that analytical tests for oil, soap, and calcium have really pin-pointed the fault and led to a solution of the trouble?

To sum up, therefore, the results of this investigation, it appears that, in the field of unlevel dyeing faults on wool piece goods, particularly velour-type cloths, there has in the past been too much emphasis on and suspicion of the chemical factors operating in the scouring, washing-off, and dyeing techniques, whereas it is now evident that physical and mechanical factors can play an equally if not more important part in the production of satisfactory results.

* * *

Since this paper was written, a follow-up experiment was carried out in which a load of pieces was scoured through an open-width aqueous scouring machine (made by F. Smith & Co., Rochdale) at 40 yd./min., soap-milled in the normal way, and then washed off in this open-width continuous machine. Subsequent dyeing to a mushroom shade gave perfect results. Further experiments on these lines with other shades are envisaged.

THOMAS RATCLIFFE & CO. LTD.
MODERNA MILLS
MYTHOLMROYD
NEAR HALIFAX
YORKSHIRE

(*Received 3rd April 1958*)

Notes

Meetings of Council and Committees
September

Council— 3rd

Finance and General Purposes— 17th

Publications— 16th

Diplomas— 17th

Fastness Tests Co-ordinating— 5th

International Federation 1959 Congress—

Scientific and Technical Subcommittee— 12th

Social Subcommittee— 29th.

Death

We regret to report the loss by death of Mr. F. H. Pollard.

Colour Index
Amendments and Additions

In conformity with the policy stated in the preamble to the *Colour Index* it is intended to publish in the January, April, July, and October issues of the *Journal* and simultaneously in the *American Dyestuff Reporter* a list of amendments and additions to the Second Edition of the *Colour Index* which have been notified to the Society during the previous three months.

The first such list, including alterations since Volume 4 went to press, appears below.

It is intended to make available to subscribers this and subsequent lists printed on one side of the page only. For further details apply to the General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

Page	*Colour Index No.*	
1297	**C.I. Acid Blue 154**	
	delete **Fenapal Blue 2R ... ...**	**G**
	delete **Palatine Fast Blue RRNA–CF**	**G**
1299	**C.I. Acid Blue 158 or 158A**	
	for **Chromacyl Wool Blue GG...**	**DuP**
	read **Chromacyl Blue GG ...**	**DuP**
2629	**C.I. Azoic Yellow 1**	
	add **Naphtazogene Yellow J ...**	**Fran**
2630	**C.I. Azoic Yellow 4**	
	add **Naphtazol Direct Yellow 2J**	**Fran**
2631	**C.I. Azoic Yellow 7**	
	add **Naphtazol Direct Yellow J...**	**Fran**
2633	*delete* **C.I. Azoic Yellow 14, 15, and 16**	
2635	**C.I. Azoic Orange 2**	
	add **Naphtazogene Orange J ...**	**Fran**
2638	*delete* **C.I. Azoic Orange 16**	
2641	**C.I. Azoic Red 1**	
	add **Naphtazogene Scarlet R ...**	**Fran**
2642	**C.I. Azoic Red 6**	
	add **Naphtazogene Red J ...**	**Fran**
2645	**C.I. Azoic Red 17**	
	add **Naphtazol Direct Scarlet R**	**Fran**
2650	*delete* **C.I. Azoic Red 39**	
2651	*delete* **C.I. Azoic Red 42 and 43**	
2662	**C.I. Azoic Blue 8**	
	add **Naphtazol Direct Blue B ...**	**Fran**
2664	*delete* **C.I. Azoic Blue 15**	
2858	**C.I. Solvent Red 49**	
	delete **D&C Red No. 19 ... ...**	**CCC, KO, NAC**

Light Fastness Subcommittee

A special meeting of the Light Fastness Subcommittee was held on 6th August 1958 for the purpose of meeting Mr. Joseph E. Norton, chairman of the AATCC Light Fastness Committee.

Mr Norton described recent work which has been done in America which is of particular interest to the British Subcommittee because the ISO Daylight Standards were used as well as the AATCC Standards. The work consisted in exposing all the standards together with a number of dyed textiles which had been found to give anomalous results in the past to daylight and to sunlight in three different parts of the U.S.A., and in fading lamps of various types including the earliest Xenotest. The results were assessed both with the Grey Scale, which enabled complete fading curves to be plotted, and against both sets of standards, which enabled correlation factors to be determined between each lamp and daylight and sunlight for both ISO and AATCC methods. The number of results obtained was so great that statistical analysis would have been impracticable without recourse to an electronic computer. An account of this work will probably appear in the Dutch textile journal *De Tex*.

Burnt-gas Fumes Fastness Subcommittee

Mainly because of objections in principle from Continental fastness-testing organisations to the use of a gas-fume fading test as described in the *Third Report of the Fastness Tests Co-ordinating Committee* (p. 73), the Subcommittee has been re-examining the test described in the *Second Report of the Fastness Tests Co-ordinating Committee* (p. 2). It seems probable that such a test, based on the use of oxides of nitrogen generated from acidified sodium nitrite, would gain wider international acceptance than one based on burnt-gas fumes. Consequently, the test described in the *Second Report* has been modified in detail for discussion at the ISO conference in Switzerland.

Course in Dyeing for Diploma in Technology (Dip. Tech.) at Huddersfield

The National Council for Technological Awards has recently recognised for the award of the Diploma in Technology two courses offered by the Huddersfield College of Technology. These are the

four-year Full-time Course in Dyeing, in which the students work in industry during the third year, spending the other three years in College, and the four-year Sandwich Course in Chemical Technology. The Dyeing Course provides a sound scientific training for the industries in which the chemistry of dyes and their application to textiles play an important part, whereas the Chemical Technology Course has a bias towards the large-scale production of organic compounds, particularly dyes and intermediates, and includes a study of Chemical Engineering. The entry qualification to these courses is a good Ordinary National Certificate or the General Certificate of Education Advanced Level in Chemistry and Physics.

It is historically appropriate that Huddersfield College of Technology, which is recognised as a Regional College, should be the first College in the country to have a Dyeing Course approved, since it was there that the first organised courses in dyeing were held in the early 1860s. George Jarmain, Dan Dawson, J. B. Wilkinson, Joe Frost, and Charles Holliday were pioneers in this work. Huddersfield has always had a strongly developed dye industry, which, with the College, has played a big part in the history of the development of dyes.

Continental (E.C.E.) Colour Fastness Tests

The fifth general meeting of E.C.E. (Groupement d'Études Continental Européen pour la Solidité des Teintures et Impressions) was held at Rotterdam during 8–9th May 1958. Eight countries—Austria, Belgium, France, Germany, Holland, Italy, Spain, and Switzerland—took part, and three members of the Society's Fastness Tests Co-ordinating Committee—Dr. P. W. Cunliffe, Mr. K. McLaren, and Mr. G. B. Angus—were present as observers; the Scandinavian countries had sent apologies.

It was reported that a new edition of the E.C.E. fastness tests (*Code des Solidités*) was being published this year in France, Germany, and Switzerland. The main subjects under discussion were the reproduction and distribution of the blue and grey scales, use of the xenon lamp in light-fastness testing, and fastness to weathering, perspiration, thermal treatments (pleating, etc.), burnt-gas fumes (the term *fastness to oxides of nitrogen* was preferred), and dry cleaning.

According to the report in *Teintex* (**23**, 497–498 (July 1958)) the E.C.E. was to be indirectly represented at the meeting of the Subcommittee ISO/TC 38/SC 1 to be held in Lucerne during September 1958.

Swiss Course in Fastness Testing

On the occasion of the bringing into force of the ISO (International Organisation for Standardisation) tests by the SNV (Schweizerische Normen-Vereinigung), the SVCC (Schweizerischer Verein der Chemiker-Coloristen) organised a course of instruction on colour fastness at the EMPA (Eidgenössische Materialprüfungsanstalt) in St. Gallen. Owing to the large number (eighty) wishing to attend, the course was held first on 15th March 1958 and repeated on 26th April 1958. In the morning Dr. Weibel outlined the history and the present position of fastness testing, while the afternoon was devoted to practical demonstrations—use of blue and grey scales, fading lamps, wet fastness, etc.

The SNV has just published a new edition of Swiss fastness tests in the form of a dossier containing a total of 45 individual tests—32 ISO recommendations, 5 ISO tentative tests, and 8 ECE tests. The position with regard to each test is listed in *Textil-Rundschau*, **13**, 231–233 (April 1958), and the tests themselves are available from EMPA, Unterstrasse 11, St. Gallen, Switzerland.

International Conference on Co-ordination Chemistry

The next International Conference on Co-ordination Chemistry will be held in London from Monday, 6th April, to Saturday, 11th April, 1959. It will be organised by the Chemical Society under the sponsorship of the International Union of Pure and Applied Chemistry. A circular giving details of the scope of the Conference, the procedure for the submission of papers, and preliminary information about registration is available from the Secretary, The International Conference on Co-ordination Chemistry, c/o The Chemical Society, Burlington House, London W.1.

Association Internationale de la Teinture Textile

The annual congress of the A.I.T.T., representing ten countries including Britain, was held in Salzburg at the beginning of June. M. Requette was elected president in the place of M. Pierre Baumgartner, who had occupied the office during the six years of the Association's existence. At the same time the headquarters of the A.I.T.T. was transferred from Paris to Ghent, the headquarters of the Belgian Association of Finishers. Reports showed that in nearly every member country the turnover in textile finishing increased in 1957 compared with 1956, but that there had been a distinct falling off since the end of 1957. There was general agreement that possible increases in productivity had been prevented largely by the often exaggerated quality requirements of customers and the infinite variety of patterns. Productivity could be increased by such measures as standardisation of lengths and widths, better packing of grey cloths, and use of water-soluble sizes (*Teinture et Apprêts*, (46), 140 (June 1958)).

The economic committee of the A.I.T.T., under the chairmanship of Dr. Guido Ziersch (Germany), was concerned with working out a common trade policy in preparation for changes in the textile-finishing trade that are likely to result from the European Common Market and possible formation of a European Free Trade Area. The A.I.T.T. asks to be consulted by the appropriate authorities in this connection. The A.I.T.T. also regards it as

essential eventually to create usable international statistics of textile finishing, which are largely lacking at present.

The technical committee discussed standard test methods for abrasion resistance, "no-iron" finishes, water-repellent and waterproof properties, and the shrink resistance of wool fabrics. Proposals were also considered for the marking of textiles from the point of view of their "cleanability", which may be influenced by the effect on coloured textiles of fluorescent brightening agents in detergents.

Verein der Textil-Chemiker und -Coloristen

The annual conference of the V.T.C.C., held in Baden-Baden during 1st–3rd May 1958, celebrated the completion of the first decade of its life. The President of the V.T.C.C., Professor E. Elöd, contrasted conditions in Germany ten years ago, just after the currency reform, when the present prosperity could hardly be foreseen among the destruction and disorganisation remaining after the 1939–1945 war. He also outlined the technical revolution brought about by the development of new fibres and new classes of dyes during this decade. Among the hundred visitors from abroad, Dr. H. Ris represented the International Federation of Associations of Textile Chemists and Colourists, and issued a general invitation to the Congress to be organised by the Society in London during September 1959. Professor R. H. Peters spoke on *The Teaching of Textile Chemistry in the United Kingdom*, and Dr. T. Vickerstaff gave a paper on *Reactive Dyes for Textiles*.

New French Legislation on Food Colorants

The *Journal Officiel* of 9th April 1958 contains an *arrêté* of 25th March 1958 amending that of 28th June 1912 governing the artificial coloration of foodstuffs. The new order, which is reproduced in *Chimie et Industrie*, **79**, 862–865 (June 1958), applies to foods manufactured after 30th September 1958. The colorants must not contain more than a trace of chromate, cadmium, mercury, selenium, uranium, polycyclic hydrocarbons, or non-sulphonated aromatic amines, nor more than 40% of diluent. The new order lists 18 groups of foodstuffs with permitted colorants, and appendixes list the colorants themselves (22 natural and 11 synthetic) with their (old) *Colour Index* numbers.

Two Centuries of Textile Education in Austria

This year the *Bundeslehr- und Versuchsanstalt für Textilindustrie* in Vienna can trace its history back for 200 years. According to O. Reiter (*Textil Praxis*, **13**, 622–623 (June 1958)) in 1750 the requirements of the Austrian imperial court and the aristocracy for fancy textiles for fashionable clothes were met almost entirely by imports, mainly from France. In order to become independent of the French designers and to encourage the domestic industry, the Empress Maria Theresia sent an expert, Florian Zeiss, to France to study the manufacture of brocades, ribbons, etc. On his return he founded the *K.K. Commerzial-Zeichnungsakademie*, which was opened on 11th January 1758 with six pupils.

In its long history the school underwent several changes of name and of address. Girls were admitted in 1910; a senior department for textile chemistry was added in 1926, and building of a testing station (*Versuchsanstalt*) for all branches of the textile industry was begun in 1929. After severe bomb damage to buildings in 1945 the school has been further developed, and in 1957 a department for "office technique" was added.

In textile chemistry the equipment now available for instruction includes, apart from scouring, dyeing, and finishing machinery, a complete screen-printing installation with photochemical preparation of screens. Courses in dyeing include a junior (*Fachschule*) course of three years, and a more advanced course of five years. Those who are successful in the examination (*Reifeprüfung*) at the end of the advanced course may, after at least four years' suitable practical experience, apply for permission to call themselves *Ingenieur*.

Textile Museum in Tilburg

The new textile museum in Tilburg in Holland was officially opened on Saturday, 24th May 1958, when there was also an exhibition entitled *Kleurenpracht van Soemba-weefsels* ("Colour in Soemba Fabrics"). The collection includes about 1500 books, in addition to various printing blocks, Dutch printed fabrics, dyeing recipes, pattern books, and oriental fabrics. Close contact is being maintained with the textile museum in Enschede.

Textile Research in Germany

The formal opening of new buildings at two textile research institutes indicates the importance now being attached to textile-chemical research in the Federal Republic. The new chemistry building of the Deutsches Forschungsinstitut für Textilindustrie in Reutlingen was opened on 17th April 1958. The close association with the Staatliches Technikum für Textilindustrie in Reutlingen will ensure that practical aspects will not be overlooked, and will provide the opportunity for trials on a semi-bulk scale, while relations with the Technische Hochschule in Stuttgart will provide more academic contacts.

In the Rhineland the new building of the Forschungsinstitut of the Textilforschungsanstalt Krefeld was opened on 30th May 1958. In the speeches emphasis was laid on the importance of applied research and of the Textilingenieurschule for the velvet and silk town of Krefeld.

Deuxièmes Journées Internationales de la Couleur—Toulouse, 19th–22nd March 1958

The aim of these conferences, the first of which was held at Amiens in May 1957, is to bring together specialists in the various activities in which colour plays a leading rôle. In the session on colorimetry, held under the chairmanship of Professor Yves Le Grand (Paris), colorimetry and spectrophotometers were discussed by F. Braun

(Louvain), Professor W. D. Wright (London), and M. Bertrand (Paris). Papers were presented also on the measurement of the whiteness of papers and the reproduction of colours in photography and in printing, and attention was drawn to various difficulties and dangers associated with colorimetric measurements. Although a paper was read on the assessment of the light fastness of dyeings, the textile industry was not well represented among the experts from Continental countries and Britain. Among other subjects discussed were the standardisation of safety colours, the teaching of colour, colour in architecture, the standardisation of printing inks, and colour in cartography. All the papers are to be published as a single volume by the organisers of the conference—the Centre d'Information de la Couleur, 23 rue Notre-Dame des Victoires, Paris (2e).

Colorimetry and its Application

According to *Textil Praxis* (**13**, (68) (Feb. 1958)) a conference on the above theme was held in Berlin during 9–10th October 1957 under the joint auspices of the *Fachnormenausschuss Farbe* (Committee on Colour Standardisation) and the *Fachausschuss Farbe des Verbandes Deutscher Physikalischer Gesellschaften* (Committee on Colour of the Association of German Physical Societies). In the *Bundesanstalt für Materialprüfung* a small exhibition was organised consisting of about forty historic, commercial, and experimental colour-measuring instruments.

The twenty lectures, at which the attendance was some 300, could be classified in three groups—methods of measurement, applications, and special problems. It was stated that in the future the Beckman spectrophotometer might be available in automatic form, and electronic methods of evaluating colour from spectrophotometric measurements were discussed; four new types of colorimeter were described. Among special problems, perhaps the most difficult is that of the white standard, the fundamental principles of which were discussed. The question of tolerances in colour specifications is also of considerable importance. Finally, the use of interference filters in colour measurement and for additive colour mixture was demonstrated and discussed.

International Committee for Cellulose Analysis

This year's meeting of the ICCA was held in Darmstadt during May 10–12th, and was attended by 34 delegates representing 13 countries. The rules of the ICCA are given in *Svensk Papperstidning*, **61**, 413 (30th June 1958), where the discussions in the various subcommittees are summarised. These were concerned with—alkali solubility, carboxyl groups, viscosity (in cuprammonium, etc.), permanganate number, lignin, pentosans, and chromatography. The present secretary of the ICCA is fil. lic. P. O. Bethge, Svenska Träforskningsinstitutet, Drottning Kristinas väg 61, Stockholm Ö, Sweden.

Dyeing, Printing, and Finishing in Hong Kong

According to a review of the cotton industry in Hong Kong (*Textile Recorder*, **75**, 58–60 (April 1958)) the dyeing and printing industries received a stimulus from the influx of technicians, machinery, and finance from the mainland of China from 1949 onwards. Thus one works underwent a fivefold increase in size between 1949 and 1957. New equipment has been installed and modern finishing processes are to be applied. While the principal markets were formerly such Far Eastern countries as Thailand and Indonesia, exports are now being directed to America and Europe. It is expected that within a few years the capacity of the industry will rise to 75 million square yards of cloth.

German Textile-finishing Industry

The total number employed is 67,300 and the turnover DM 531 millions, showing an increase of 4·6% for 1957 compared with 1956 (*Melliand Textilber.*, **39**, 942 (Aug. 1958)). Investment at DM 16 million was below the required level. Productivity could be raised if customers could decide on the following—(*a*) standardisation of piece lengths, (*b*) standardisation of width, (*c*) uniformity of packing, (*d*) use of water-soluble sizes, and (*e*) avoidance of unsuitable additives, in view of the steadily increasing importance of durability in goods.

Terminology of Fibres

Skinner's Silk and Rayon Record reports (**30**, 547 (May 1958)) that German firms, with the European Common Market in mind, have suggested as a basis for discussion a systematic terminology for synthetic fibres, which would at once indicate the nature of a fibre with an unfamiliar trade-name, e.g. Diolen. The prefix "poly-" would be omitted as pleonastic; the chemical nature of the fibre would be indicated by two capital letters, e.g. AD = (poly)amide, GT = glycol terephthalate; and the type of fibre (filament or staple) would be mentioned. Thus we might have e.g. *Diolen GT staple*.

New Soviet Textile Journal

The first number of *Tekhnologiya tekstil'noĭ promÿshlennosti* ("Technology of the Textile Industry") has just been published by the Ministry of Higher Education of the U.S.S.R. The new journal is intended to foster scientific and technical progress and higher productivity, and will report work carried out in the technical colleges of the U.S.S.R. It will contain also abstracts of the most important work carried out in the U.S.S.R. and abroad, and reviews of monographs and textbooks. It will appear every two months.

New Books and Publications

Deutscher Färberkalender 1958

Edited by O. Mecheels. Pp. iv + 64 + i + 440 + 64 + 59. Munich: Franz Eder Verlag. Price, DM 13.00.

The 62nd issue of this well known yearbook follows in all respects the style and contents of previous years, and fully maintains the high standard that it has set in the past. In addition to the usual reference sections— lists of German man-made fibres, new dyes, and auxiliary agents (mainly of German origin, ICI being the only British firm mentioned), etc.— there are some thirty articles, some original, while others are simply accounts by technologists on the staffs of various dye and chemical makers of the application of some range or other of their products, e.g. *Dyeing of Trevira with Intramin Dyes, Dyeing of Polyester Fibres and their Blends with Cotton with Indanthren Dyes*, etc. There is a useful article on the *Latest Methods of Continuous Chlorite Bleaching* by H. Wagner (pp. 33–62) and one on the *Identification of Fibres and Dyes* by A. Bode (pp. 93–102). Now that the number of pages in this yearbook has become so large, so much so that it can no longer be regarded as a book to carry in the pocket but solely as a book of reference to have ready on the bench or desk or, most probably, in the library, the publishers should consider increasing the size of the page, as there is no doubt that this would make the book much handier to use. To do so might be breaking with a long established tradition, but tradition should not be allowed to impede usefulness. If the change were to be made and the present high standard of its contents maintained, in a very few years a new tradition would be established to replace the old one.

C. O. Clark

Symposium on Trade Wastes

London: Institute of Sewage Purification. 1957. Pp. 112 + 55 (*Report of Discussions*). Price, 10s. 0d.

The papers presented at the above symposium, organised by the Midland Branch of the Institute and held at the University of Birmingham on 9th April 1957, should be read by all who have to do with the disposal of trade wastes.

The first two papers deal with the problem as seen by the authorities most concerned, namely the Sewerage Boards and the River Boards. Recent legislation in the River Boards Act 1948 and the Rivers (Prevention of Pollution) Act 1951 was introduced with a view to strengthening the power of such bodies. Whilst it is fairly readily appreciated that waste liquors discharged into streams or rivers should be free from noxious bodies, it may surprise many to know that, before discharge may be made into a sewer of a local authority, the effluent must conform to certain high standards, and considerable obligations are imposed.

Perhaps the most interesting paper to members of the Society is that which describes the effluent treatment processes carried out at some five factories including a textile dyeing and finishing works and a rayon factory. The remaining papers deal with the treatment of such specific wastes as arise from metal-plating and gas works. The textile chemist employed in a textile works of average size may read these and be thankful that the problems are not his.

The symposium finds no ready-made remedy, either in the papers or in the discussion, but serves to illustrate how the problem may reach a satisfactory solution only by hard work, much consultation, and also the spending of much money. The last point, alas, is not readily appreciated by many administrators, who may find money to ensure a good-quality supply of water but are less ready to do so for disposal after use. They, too, will require educating.

L. Morris

Emulsions: Theory and Practice

By Paul Becher. Pp. ix + 382. New York: Reinhold Publishing Corpn. (London: Chapman & Hall Ltd.). 1957. Price, $12.50 (100s 0d.).

In the preface the author states that he has, with few exceptions, consulted all the original sources. That this statement should have been necessary lends support to the reviewer's opinion that American scientific and technical textbooks are compiled from the abstracts, without reference to the original papers.

The present work is sponsored by the American Chemical Society, and is the 135th in its monograph series. It is divided into nine chapters and two appendixes, and there is a subject index. After a brief introduction the next two chapters deal with surface phenomena and the physical properties of emulsions; then follow two chapters on emulsion stability and instability. Chapter 6 contains a brief summary of the chemical nature of emulsifying agents, and the next chapter contains a survey of the techniques and apparatus for making emulsions. Chapters 8 and 9 deal with the treatment of commercial emulsions. Appendix A contains a discussion of the measurement of emulsion properties and in Appendix B there is a list of the commercial emulsifying agents available in the U.S.A.

In general the immense and confused subject-matter is well treated, and the reader is soon warned of the dangers of facile acceptance of some of the more futile hypotheses that have been propounded. Dr. Becher is to be congratulated on finding a happy balance between conjecture and theory. The only significant omission is a discussion of the importance of liquid crystals in many so-called emulsions.

Printing and production of the book are good. It is a pity that the text is marred by too generous a sprinkling of typographical errors. For example, on pp. 86–87 the reviewer noticed three in as many

paragraphs; Fig. 2.19 is said to show a dotted curve; and the word "data" is used as both singular and plural. English readers will scarcely approve of expressions such as "helter-skelter" for *randomly*, "hypothesized" for *postulated*, and "eventuated" for *was formed*, and they will certainly shudder at such phrases as "no single simple structure will be found to satisfy all the phenomena" (p. 43) and "The very antiquity of emulsion formulation is a hinderance to postulating a scientific theory of emulsions." (p. 85).

All chemists interested in emulsions, provided that they can ignore the irritations of typographical errors and linguistic vagaries, will find this a useful refresher course and reference book. The price, however, will place the book beyond the reach of most workers.

E. V. Truter

Anorganische Pigmente und Röntgenstrahlen

By R. König. Pp. xi + 132. Stuttgart: Ferdinand Enke Verlag. 1956. Price, DM 27.00.

This book opens with a brief introduction in which the author notes the classes of crystal symmetry and the Bravais lattices, after which three pages are devoted to crystal chemistry, four to the production of X-rays, five to the Debye–Scherrer powder photograph, and six to some rather general remarks about the amorphous and crystalline states of matter. In the space of only 25 pages it is obviously not possible to do much more than mention most of the topics and to refer the reader to suitable books. The dangers of excessive condensation are well known, and it is questionable whether an introduction of this length would be of much value to a newcomer to this subject. Erroneous statements, such as the remark that the highest possible coordination number is 12, could be avoided by a little further amplification.

The main section of the book consists of some 80 pages of systematically arranged data and literature references to X-ray work on coloured inorganic compounds which are or have been used as pigments. The primary classification is based on colour and the secondary classification according to the groups of the Periodic Table. The X-ray data listed are dimensions and content of unit cell and space group. The book concludes with some 20 pages of photographic reproductions of X-ray powder photographs.

In a book published in 1956 one might expect to see some mention of focussing powder cameras and some reference to the use of Geiger-counter spectrometers. The industrial worker is interested in identification, including the detection of impurities and the recognition of polymorphism, and in the degree of crystallinity of a pigment; he does not normally want unit cell dimensions or space groups. It is difficult to see what use the reader can make of reproductions of powder photographs: if powder data are to be included in a book, it would be preferable to give the spacings and relative intensities of the diffraction lines. No reference to the widely used A.S.T.M. index of powder data was noticed.

The author's stated intention was to produce a small handbook embodying the results of X-ray studies of inorganic pigments which would be of value as a reference book to workers in the pigment industry. This book could conceivably be useful to someone about to start fundamental research and wishing to find out what structural work had been done on a compound but not having at hand the standard reference works from which Dr. König has compiled this volume. It is less likely to be useful to a research worker concerned with the day-to-day work in an industrial X-ray laboratory.

A. F. Wells

Zur Dichtebestimmung von Fasern

Methoden und Beispiele der praktischen Anwendung

By J. Juilfs. (*Forschungsberichte des Wirtschafts- und Verkehrsministeriums Nordrhein-Westfalen* No. 381). Pp. 65. Cologne: Westdeutscher Verlag. 1957. Price, DM 17.00.

This book is one of a series of research reports, each of which is a detailed monograph in a narrow field, but collectively the series covers a wide area. Since the war more than four hundred of these monographs have been published on many topics, about 8% being on the science and technology of textiles.

In his conclusion the author says that his purpose was to show the importance of a knowledge of the density of fibres, which, he claims, is as important a characteristic as swelling, strength, dye uptake, and X-ray diffraction. He illustrates his theme by examples showing the relations between density and swelling, between density and dyeing, and between density and stretching. In addition, he discusses how density is related to the degree of crystallinity of various fibres. In another section he discusses the changes of density which result from moisture absorption. This, and other subjects, are illustrated by graphs and tables.

A chapter is devoted to experimental methods of determining density. There are sections on the pyknometer, buoyancy, and filtration methods, with a final section on a new apparatus for the last named method. Another chapter is concerned with the apparent changes of density with time, the scatter of results, and the effects of moisture uptake. The fourth and longest chapter has ten sections, each devoted to the relation of density to some other parameter of fibres.

In general, the subject-matter is presented clearly, and there is a short but useful bibliography. The line drawings are clear, but the half-tones have suffered in reproduction.

Most of the subject-matter has already been published, but its collection under one cover will be found useful. The monograph can be recommended as a starting point for anyone beginning work in this field.

J. M. Preston

Moderne Wasch- und Reinigungsmittel
Heft II/1
Waschen und Reinigen
Die Waschwirkung und ihre Prüfmethoden
1. Nachtragsband
mit Anschluss-Literatur 1951–1954

By H. Manneck. Pp. i + 52. Garmisch-Partenkirchen: Moser-Verlag, Fachliteratur Ermittlungs- und Berichtsdienst. 1955. Price, DM 20.00.

This is the first supplement to the monograph on washing and cleaning and the effect of washing and test methods, first published in 1952. As before, the author divides the subject into three sections—chemistry and physics of washing, testing methods and test apparatus, and evaluation and examination of detergent effect.

In all three sections the author quotes recent publications and literature references, and the monograph is an interesting compilation. There is a detailed description of various test methods for wetting, foaming, soil dispersion, lime soap and water hardness tests; to the chapter on test washing machines the new Wash Wheel (C.P.A. and S.D.C.) should be added. The author mentions also various suggestions for compounding artificial soiling matter, and notes that the universal artificial soil composition has yet to be found. Optical and photometric cleansing test methods are dealt with, also the washability of synthetic fibres, alone or in mixtures with natural fibres.

At the end is a comprehensive list of 176 references, some of which have already been quoted in other publications and books (Niven, Harris, Stüpel, Schwartz–Perry, etc.). More critical comment and guidance by the author on washing test methods would have been welcome.

A. F. KERTESS

New Books received

List of Periodicals abstracted by Chemical Abstracts with Key to Library Files and other Information. CHEMICAL ABSTRACTS SERVICE. Washington: American Chemical Society. 1956. Pp. 314. $5·00.

Produits Tensio-actifs. Vocabulaire des Surfactifs ou Agents de Surface. Norme Française T 73–000. Paris: Association Française de Normalisation. Sept. 1957 (Corrected Jan. 1958). Pp. 4. 175 francs.

Anfang und frühe Entwicklung des Zeugdrucks in der Schweiz unter besonderer Berücksichtigung des Kantons Glarus. U. FERD. BLUMER. St. Gallen: Verlag Zollikofer & Co. A.G. [1956.] Pp. 53. Swiss francs 6.25.

The B.D.H. Book of Organic Reagents. 10th edition 1958. Poole, Dorset: The British Drug Houses Ltd. Pp. vi + 182. 18s. 0d.

Annual Reports on the Progress of Chemistry for 1957. Volume **LIV**. London: The Chemical Society. 1958. Pp. 445. 40s. 0d.

Printed Textile Design. TERENCE CONRAN. London and New York: The Studio Ltd. 1957. Pp. 95. 25s. 0d.

200 years Geigy. Basle: J. R. Geigy AG. [1958.] Pp. 127.

Statistics of Production of Chemical Fibres in Japan. JAPAN CHEMICAL FIBRES ASSOCN. 1958. Pp. i + 6. (reprinted from *Japan Chemical Fibres Monthly*, **11**. June, 1958).

Principles of Cotton Printing. D. G. KALE. Bombay State: Publication Committee for Professor D. G. Kale's Book on Cotton Printing. [1957.] Pp. xix + 452 + xxii plates. 25 rupees.

Anorganische Pigmente und Röntgenstrahlen. R. KÖNIG. Stuttgart: Ferdinand Enke Verlag. 1956. Pp. xi + 132. DM 27.00.

Ververij-Beginselen en Onderwerpen die daarbij aansluiten. J. NUNNIKHOVEN. Enschede: Stichting "De Tex". 1958. Pp. 255. 14.75 florins (bound).

The Man-made Fibres Industry. R. ROBSON. London: Macmillan & Co. Ltd. 1958. Pp. viii + 135. 21s. 0d.

Zur Nomenklatur der Textilhilfsmittel, Lederhilfsmittel, Gerbstoffe und Waschrohstoffe. Beitrag zur Bereinigung der Nomenklatur unter Berücksichtigung des neuen Zolltarifes und des Warenverzeichnisses für die Aussenhandelsstatistik. W. SCHEER. Frankfurt: Verband Tegewa e.V. Jan. 1958. Pp. 53.

Small Firms with Big Problems. London: Department of Scientific and Industrial Research. [1958.] Pp. iii + 24. Gratis.

Problems in Wood Chemistry. UNITED NATIONS FOOD AND AGRICULTURE ORGANISATION and ISRAELI MINISTRY OF AGRICULTURE. Jerusalem: Weizmann Science Press of Israel. 1957. Pp. x+137. $6·00.

Knižnica L'udového Umenia. Svázok 2. L'udová Modrotlač na Slovensku. JOZEF VYDRA. Bratislava: Tvar Výtvarné Nakladatel'stvo. 1954. Pp. 207 (including 165 plates). 52 kčs.

The Use of Emulsions in Textiles (with emphasis on coloration). R. WILLIAMSON. (F.S.D.C. Dissertation). Bradford: Society of Dyers and Colourists. [1958.] Pp. 66. [Mimeographed.]

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

New Fabric-dyeing Machine

P. A. Mel'nikov

Tekstil. prom., **18**, 39–41 (May 1958)

A fairly detailed description of a fabric-dyeing machine (rope form) ZhM–260–L, equally suitable for washing and impregnating, is given. Using spiral sequence and controlled tension, this machine, which works at a speed of 90 metres/min., requires less space and electric energy than a multiple-box machine. G.J.K.

PATENTS

Pelleting of Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. *USP* 2,807,523

Carbon Black (C.I. Pigment Black 6 and 7)

Phillips Petroleum Co. *USP* 2,809,098

A method of removing the coke from inside an oil feed nozzle to a carbon black furnace without removing the tube from the furnace and without shutting the furnace down. C.O.C.

Extracting Water from Loose Wool or the Like

Sanderson & Murray *BP* 799,438

The wool is placed beneath a deflated flexible bag and the bag inflated. A press incorporating this principle is described. C.O.C.

Drying Carded Laps

Fleissner & Sohn Maschinenfabrik *BP* 799,832

Drying Staple Fibre

American Viscose Corpn. *BP* 799,960

Drying Fibres obtained by Chemical Retting

Patenthandels *BP* 797,605

The fibres do not stick together on drying if they are first washed free from chemicals, and then the water is expelled by use of a water-miscible solvent, which is finally evaporated off. The whole process is carried out in a hermetically sealed plant. C.O.C.

Continuous Processing of Thermoplastic Yarns

Universal Winding Co. *USP* 2,803,105
USP 2,803,108–9

Apparatus for processing polyamide, polyester, etc. yarns by heating with incidental winding, twisting, twisting-untwisting, plying, coning, copping, rubbing, drying, dyeing, coating, singeing, sizing, etc. C.O.C.

Treating Fibres with Liquid in a Closed Circuit

P. S. Riba *BP* 798,990

A cylindrical vessel closed at both ends one of which can be opened to put yarn or fabric into the vessel, has a central tube down its length around which the material to be dyed is placed. The vessel is mounted vertically on hollow trunnions half way along its length. The mounting is such that the vessel can revolve about the axis formed between the two trunnions. These trunnions communicate with conduits outside the vessel and which open at the end of the vessel opposite to the entry for the goods. One of the conduits opens directly into the central tube while the other opens directly into the main space of the vessel. This provides a continuous path for liquid passing through one trunnion to pass through the central tube into the body of the vessel and then to the other trunnion. Dyeing is carried out by circulating dye liquor through the system while the cylinder is revolving. C.O.C.

Laboratory Dyeing Machine

Horsfall Engineering Co. *BP* 798,905

A vertically reciprocable shaft has a head from which hang yarn supports. As the shaft moves up and down, yarn on the supports is likewise moved up and down in individual dyebaths. C.O.C.

Warp Dyeing Machines

British Cotton & Wool Dyers' Assocn. *BP* 797,538

A warp dyeing machine in which the warp is not under tension and which allows it to be longer in the liquor consists of a tank divided into compartments by longitudinal partitions. A number of warp drafts are fed into one end of the tank and withdrawn at the other, each draft lying loosely in the compartment to which it is fed and from which it is withdrawn. Dye liquor is fed into the same end of the tank as the warp and withdrawn at the other end. C.O.C.

Ager

R. P. Higginbottom *BP* 797,541

Modification of *BP* 779,149 (J.S.D.C., **73**, 517 (1957)). Heated rollers placed in the air-free steaming chamber are staggered with regard to the heat exchange units so that the printed cloth etc. can pass continuously and alternatively over these units. The heat exchange units are in the lower part of the chamber with the control sections below 212°F. producing moisture for the chemical reaction while the heated rollers are at $> 212°$F. C.O.C.

Driving Mechanism for the Rolls of a Raising Machine

David Gessner Co. *BP* 799,281

Worker Roller Raising Machine

Établissements Albert C. Scholaert *BP* 799,451

An automatic device for indicating the "intensity of scratching", i.e. the relative speed of the card clothing fillets with respect to the fabric, irrespective of the speed at which the machine is being run and at which the fabric is travelling. C.O.C.

Ancillary Piercing Roll for Use with Calenders

DuP *USP* 2,801,439

An auxiliary piercing roll for use with calenders to eliminate formation of bubbles and blister streaks in coatings calendered on cloth and other flexible substrates. Uniform pressure is provided between the ends of the piercing elements and the film mounted on the calender roll, this pressure being self-adjusting, e.g. by the piercing elements being spring loaded. C.O.C.

Automatic Roller Lifting Devices for Calenders

Joh. Kleinewefers Söhne *BP* 799,416

Screen Printing of Hose and Half Hose

E. G. Harding *USP* 2,803,191

Apparatus for Coating Paper

Bowater Research & Development Co. *BP* 799,147

The surface of the applicator roll is made from a resilient material, e.g. natural or synthetic rubber having a Pusey and Jones hardness ($\frac{1}{4}$ in. ball) of 35–70. The hardness of the surface of the gate roll (70–110) is greater than that of the smoothing and spreading rolls (190–270). Both smoothing and spreading rolls are provided with means whereby their contact pressures on the applicator roll may be adjusted. A backing roll, in contact with the applicator roll, may be in the form of another applicator roll so that the web can be coated on both sides. R.A.

Drying of Printed or Coated Cloth, Paper and the like

Huck Co. *USP* 2,801,847

An arrangement whereby the suspension curve of the printed or coated material is accurately controlled at a large number of points along its path through a drying chamber, while supported by and during contact maintained with only the uncoated surface of the material. C.O.C.

Garment Presses

Jaxons (Hillington) *BP* 799,664

Dry-cleaning Today (VII p. 717)
Raising of Wools (X p. 720)
Heat Treatment of Glass Fibre Fabric coated with a Thermoplastic (X p. 723)

II—WATER AND EFFLUENTS

Biological Oxidation of Sugar-based Detergents

P. C. G. Isaac and D. Jenkins

Chem. and Ind., 976–977 (2 Aug. 1958)

Preliminary experiments indicate that detergents

derived from sucrose are less resistant than tetrapropylenebenzenesulphonate to biological breakdown in sewage treatment. W.K.R.

PATENTS

Clarifying Water
Dow Chemical Co. *BP* 799,621

A mixture of an acrylamide polymer and a bentonite slurry is a good coagulant for removing suspended matter from water. C.O.C.

Clarifying Water
BASF *BP* 799,873

Sedimentation of coarse and colloidal dispersions is accelerated by use of sulphonated anionic aminoplasts, e.g. the product obtained by condensing urea with HCHO in presence of H_2SO_3. C.O.C.

III— CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Chelating Agents. II— *trans*-1:2-Diaminocyclopentane(and butane)-*NNN'N'*-tetra-acetic Acid
V. G. Yashunskii

J. Gen. Chem. U.S.S.R., **28**, 1056–1059 (April 1958)

The preparation of the title complexing agents is described, and the apparent dissociation constants and stability constants of the corresponding Ca chelates are given. G.J.K.

Sulphonation with Sulphur Trioxide— High-boiling Alkylated Benzene
E. E. Gilbert and B. Veldhuis

Ind. Eng. Chem., **50**, 997–1000 (July 1958)

Oil-soluble sulphonates are derived from high-boiling alkylated benzene (a mixture of mono- and di-alkylated benzene), a by-product in the manufacture of dodecylbenzenesulphonate, known as polydodecylbenzene (PDB). The relative merits of SO_3 vapour and 20% oleum for sulphonating PDB from five sources are reported. The former gives better yields, less odour, and lower inorganic salt content, with quicker processing time, whilst the latter provides a better coloured product, and lower heat of reaction. W.K.R.

Arsenic in Detergents
J. M. A. Lenihan, H. Smith, and J. G. Chalmers

Nature, **181**, 1463–1464 (24 May 1958)

High As contents (42 p.p.m.) found in human hair were shown to be due to washing with a particular liquid synthetic detergent containing As (ca. 80 p.p.m.) as marketed. Six other commercial synthetic detergents examined contained negligible As (0·5–8·5 p.p.m.). The As in the first product was traced to the use, in its manufacture, of sulphuric acid made by the lead chamber process from pyrites containing As. This detergent is now prepared from sulphuric acid derived from sulphur, and the As content is < 2 p.p.m. The high As content of the hair was attributed to washing with undiluted detergent and inadequate rinsing. W.K.R.

Phase Relations and Specific Salt Effects in Soap Solutions
T. Nash

Chem. and Ind., 590 (17 May 1958)

A dilute soap solution forms a two-component system, partially miscible in the liquid state (solution and micelles), the solid phases being soap crystals and ice. It should therefore have an "invariant point", i.e. a temperature, not dependent on concentration, at which both crystals and micelles are present together. The invariant point was determined for cetyltrimethylammonium bromide and cetylpyridinium bromide, by fluorescence measurements after the addition of a fluorescent dye of opposite charge, and the effect of salts thereon was noted. W.K.R.

Chemistry of Condensation of Commercial Urea-Formaldehyde Resins
Z. Wirpsza

Przemysl Chem., **37**, 38–43 (1958): *Chem. Abs.*, **52**, 12452 (25 July 1958

Studies of the condensation of urea and HCHO (at ratios 1:1·9, 1:2·1 and 1:2·5) were carried out by determining the concentrations of free HCHO, methylol radical, intermolecular bonds and behaviour of the resins to water. When an alkaline solution of urea in HCHO is boiled, the pH falls rapidly from 7·2–8·0 to 6·1–6·6 and sometimes even to 5·7–5·8. At the start mono- and dimethylolurea are mainly formed. The number of methylol radicals increases rapidly because of addition reactions (simultaneously with numbers of intermolecular bonds). The speed of formation of methylol radicals increases when the ratio of HCHO to urea increases. After reaching a maximum the number of methylol radicals begins to decrease because of condensation. The addition reaction is $-NH_2 + HCHO \rightarrow -NHCH_2OH$ and the condensation reaction $-NHCH_2OH + -NH_2 \rightarrow -NHCH_2- + H_2O$ (and to a certain extent $-NHCH_2OH + HOCH_2NH \rightarrow -NHCH_2OCH_2NH + H_2O$). During continued condensation the number of intermolecular bonds increases mainly because of decrease of HCHO in the methylol radicals. Free HCHO then starts to increase slowly and may even become 10% of the total HCHO. C.O.C.

PATENTS

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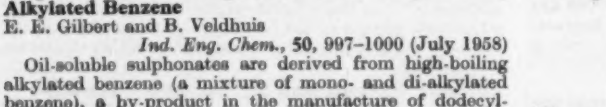
Dow Chemical Co. *USP* 2,806,060

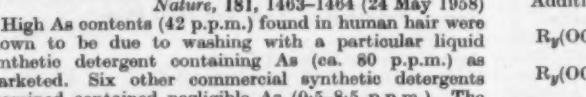
Compounds of formula—



(X = a bivalent low mol. wt. alkylene radical putting 2–3 C atoms between the N atoms; R = CH_2COOM or CH_2CH_2COOM (M = H, alkali metal, NH_4 or substituted NH_4)), e.g. *N*-(2-hydroxy-3-iminodiethanol propyl)-*NN'N'* ethylene diamine triacetic acid, are good chelating agents for bi- and trivalent metal ions in strongly acid to strongly alkaline aqueous liquors. C.O.C.

Improving the Properties of Soap
Armour & Co. *BP* 799,279

Addition of a compound of formula—



($x = 0$–25; $y = 0$–50; R = CH_2CH_2OH or C_3H_6OH; total mol. wt. = 1500–6500)
(i.e. the Tetronics of Wyandotte Chemicals Corpn.) added to soaps increases their cold water solubility. C.O.C.

Sizes for Cellulosic Warps
Monsanto *USP* 2,806,020

The alkali metal salts of low mol. wt. copolymers of vinyl acetate (90–98%) and an unsaturated compound of 3–9 C and containing at least one carboxyl group, e.g. crotonic acid, are excellent sizes for warps except those of nylon. C.O.C.

Size for Hydrophobic Yarns
Rohm & Haas Co. *USP* 2,807,865

Ammonium or alkali metal salts of copolymers of 5–20 mol. of acrylic or methacrylic acid and 95–80 mol. of an acrylate or methacrylate of an aliphatic alcohol of 1–18 C, and having a Knoop hardness of < 7 (as determined by the procedure described in *Indentation Hardness Testing* by V. E. Lysaght (Reinhold Publishing Co.) p. 189) as excellent sizes for nylon and similar hydrophobic fibres. They are readily removed during scouring. C.O.C.

Yarn Lubricant
Eastman Kodak Co. *USP* 2,807,864

In making loopy or bulk type yarns from man-made fibres a lubricant made up of mineral oil, a mahogany sulphonate, a terpene ether of the type described in *USP* 2,136,011, a long chain aliphatic acid and an alkylol amine, enables a greater amount of looping or bulking to be produced and so looks the loops as to resist pulling out even at the stress required to break the yarn. C.O.C.

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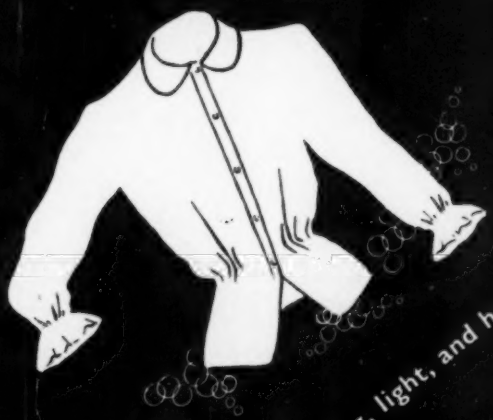

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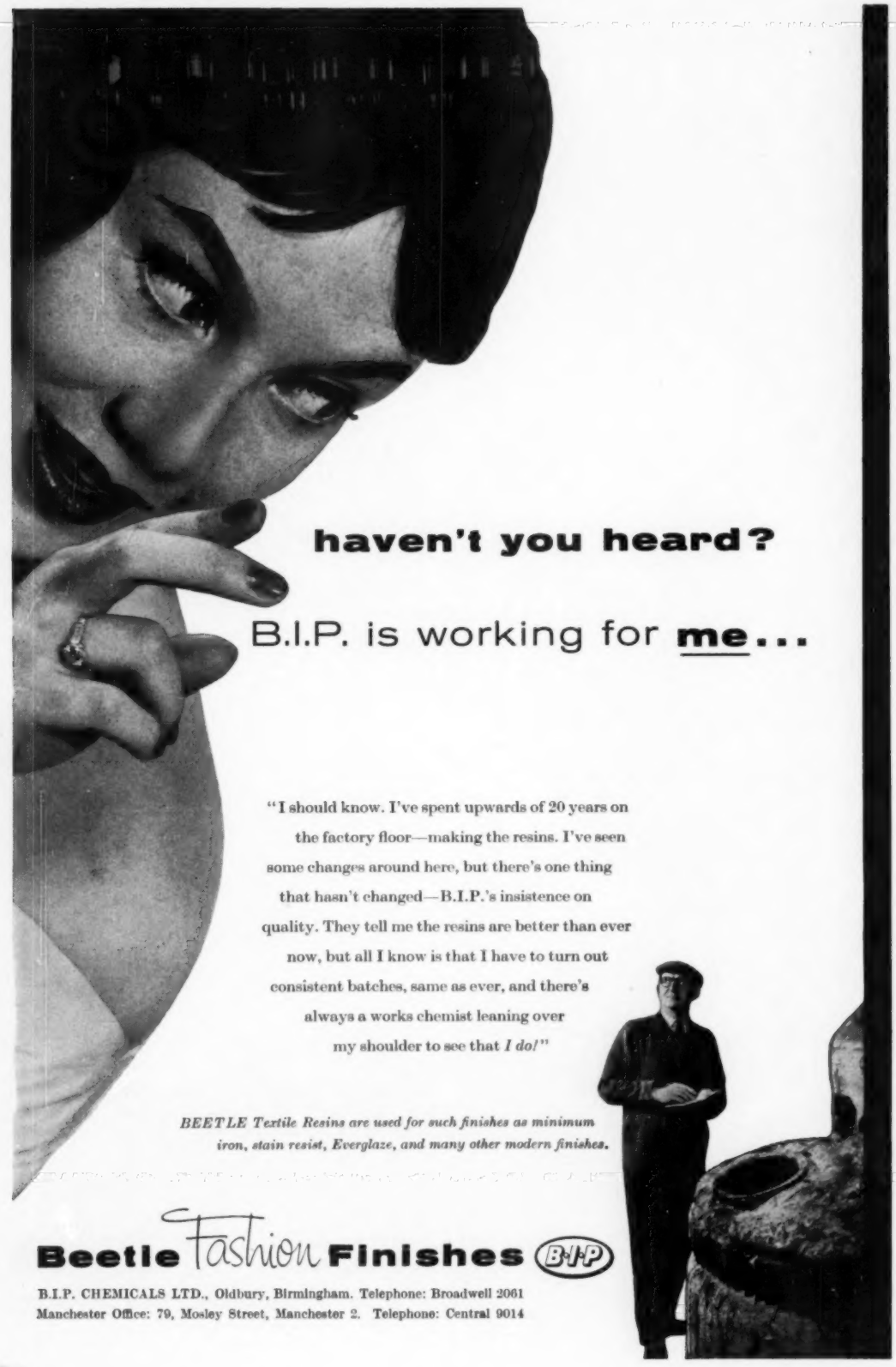

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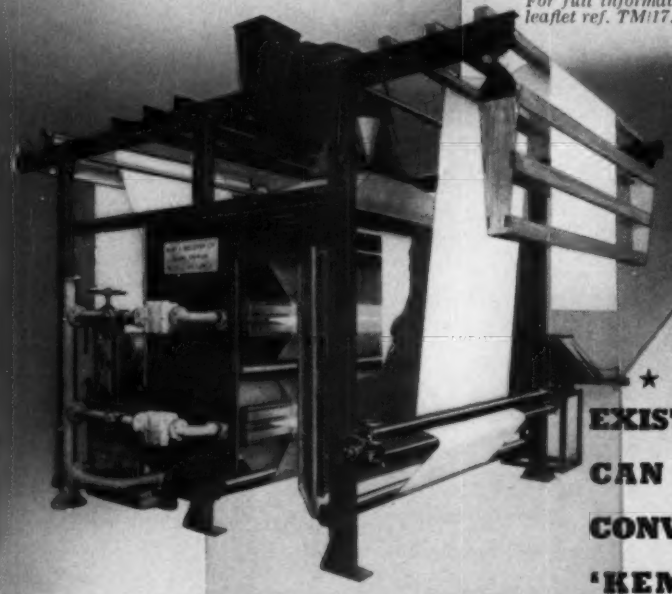

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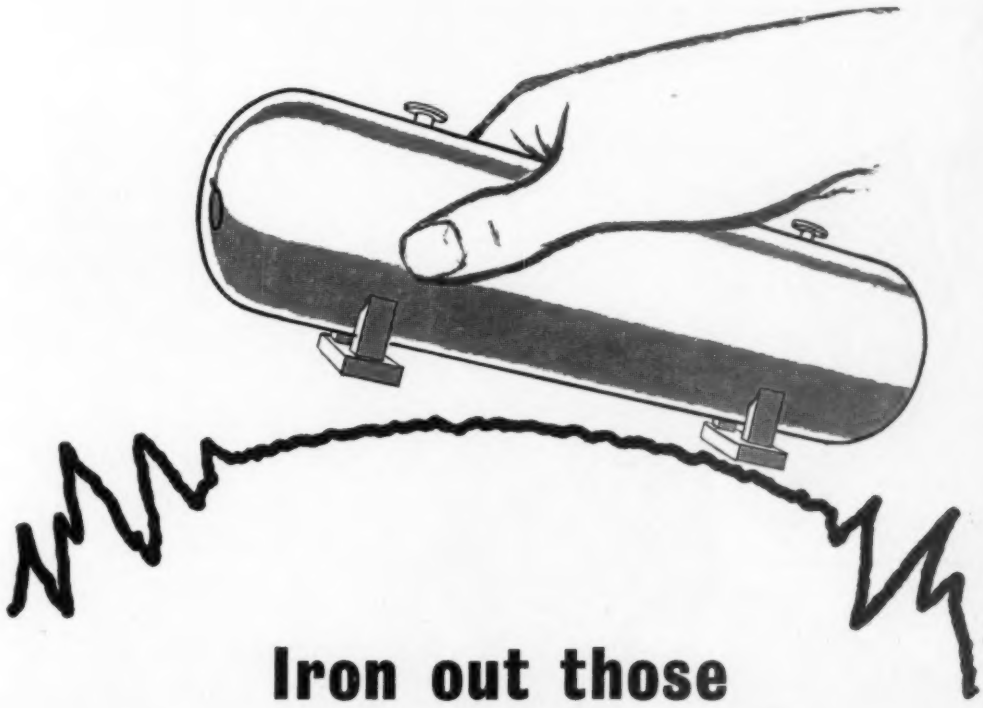

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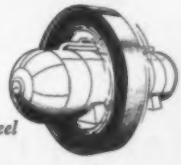

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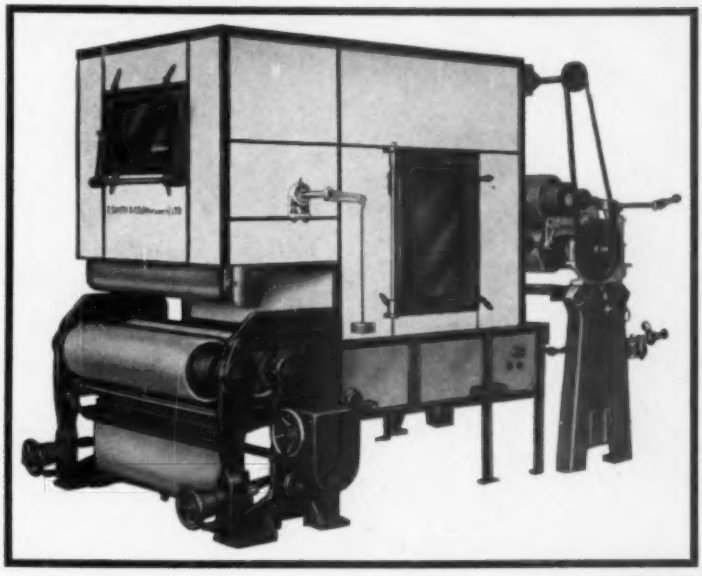

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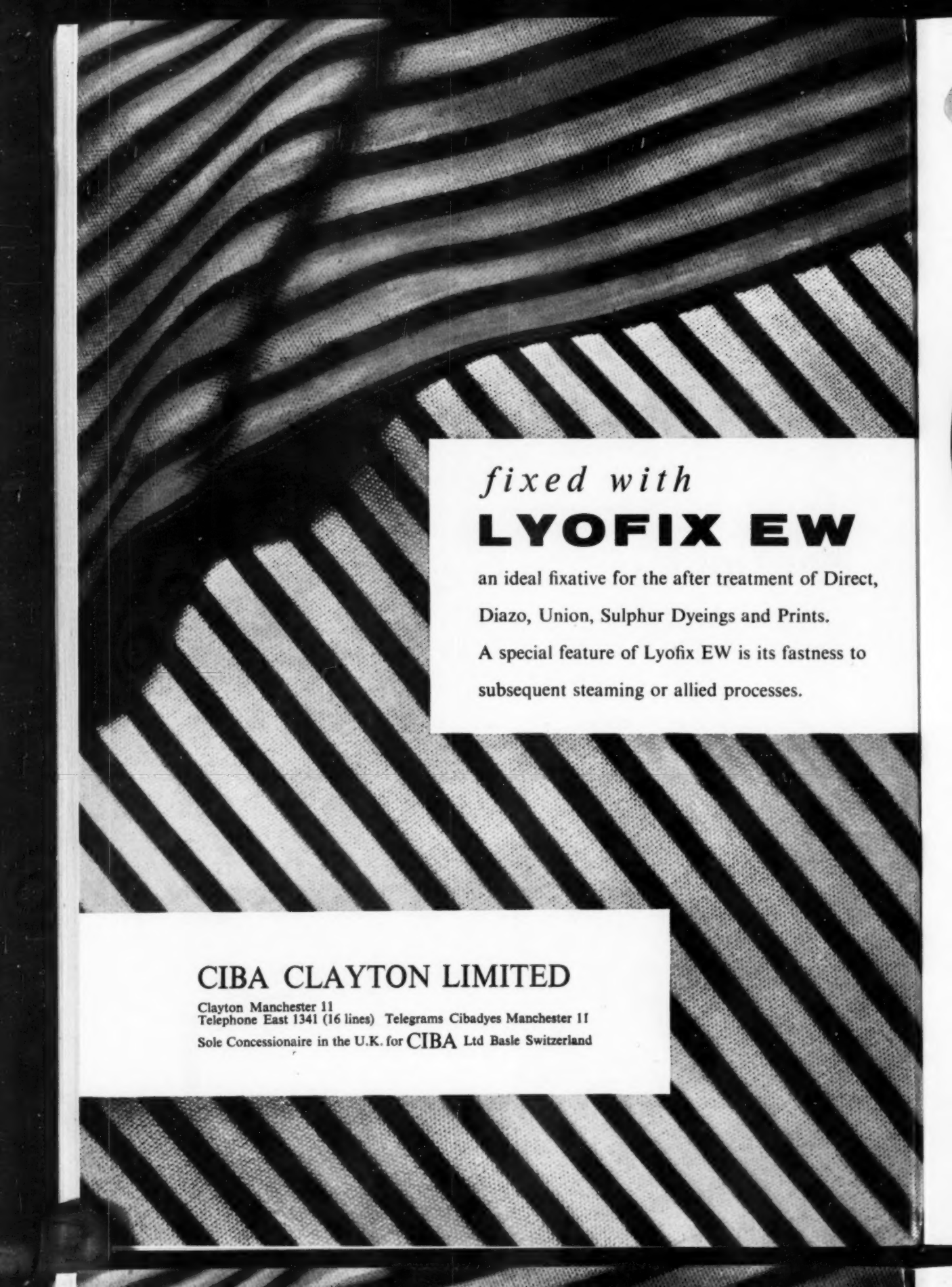

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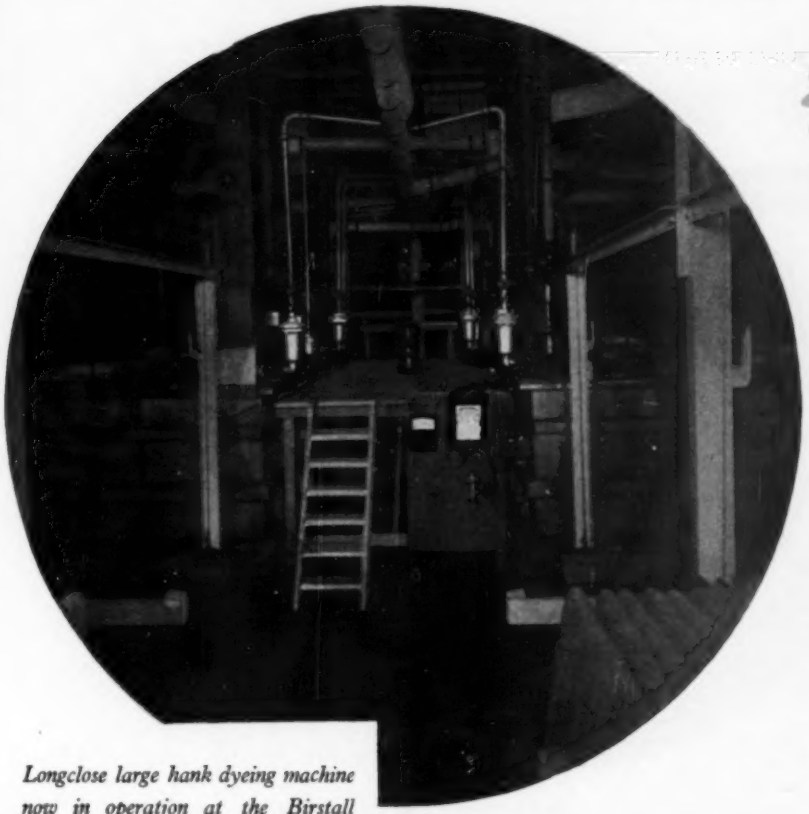

Longclose large hank dyeing machine now in operation at the Birstall Carpet Co Ltd, for dyeing 9,000 lbs of yarn at a time. 12,500 gallons of liquor are withdrawn, mixed and interchanged between four tanks and temperature control is a critical feature. Any slight difference in the rate of temperature rise between the tanks would cause lack of uniformity of shade. The machine is fully automatic: the temperature of the four vats is controlled by a Drayton D.V. 3 Dye Vat Control.

DRAYTON dye vat controls, for all types of dyeing machines, are completely automatic, absolutely dependable and do not call for skilled operators. They maintain a continuous degree of accuracy unobtainable by manual control. They reduce working costs and ensure improved and uniform production. For full information write for the booklet "The Application of Automatic Control to the Dyeing Process".

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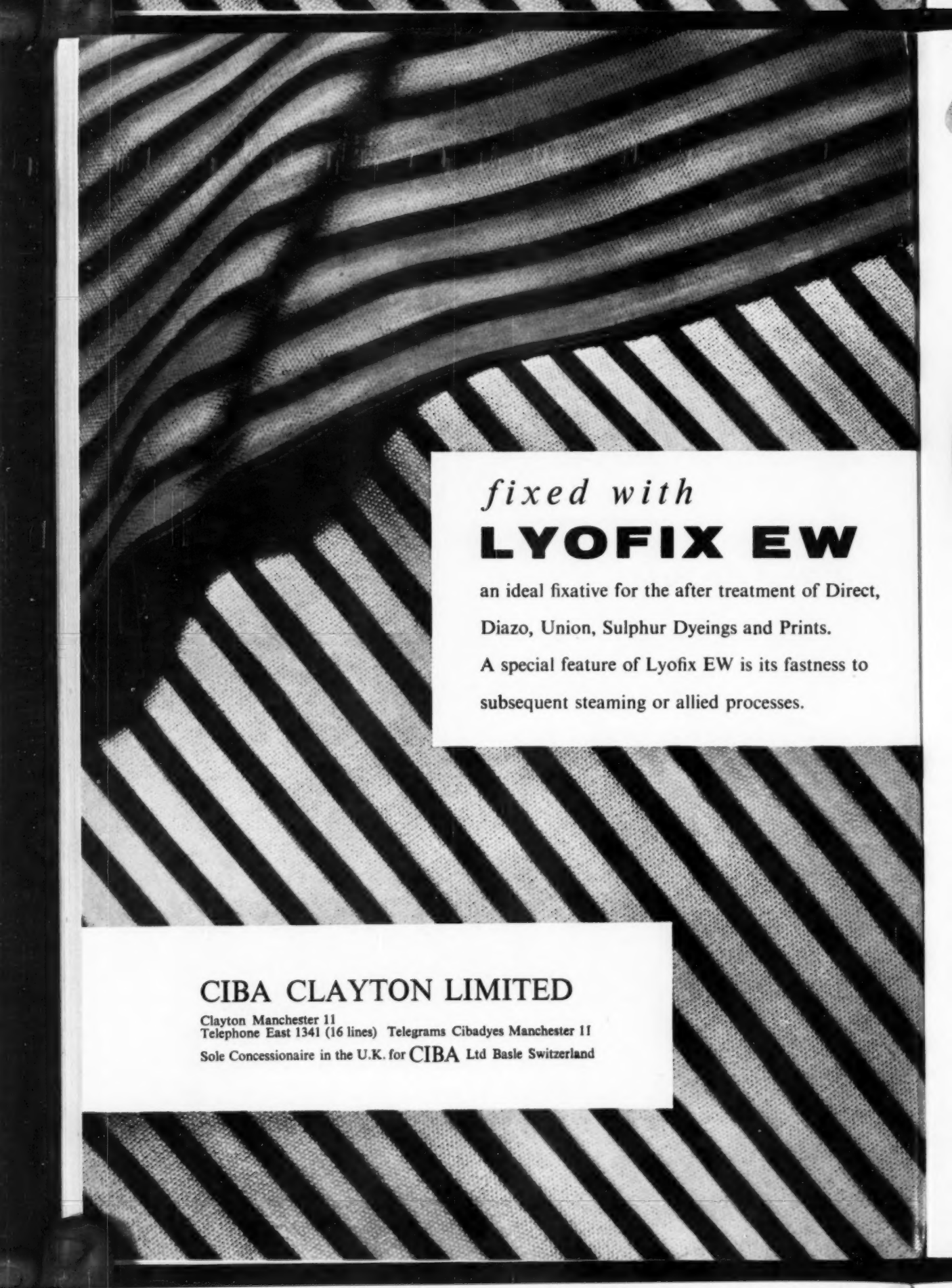

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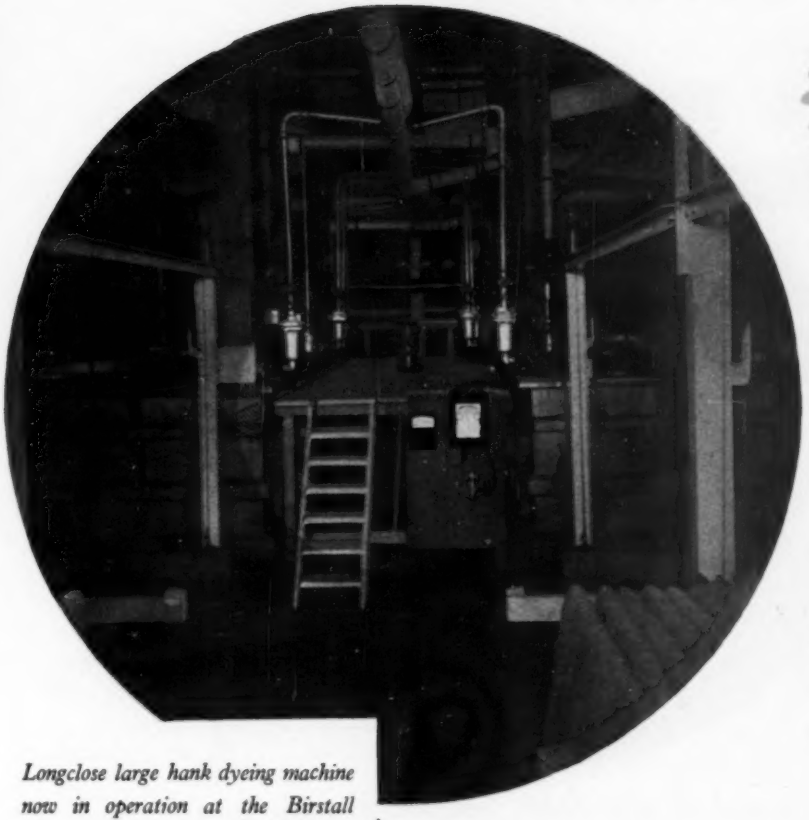

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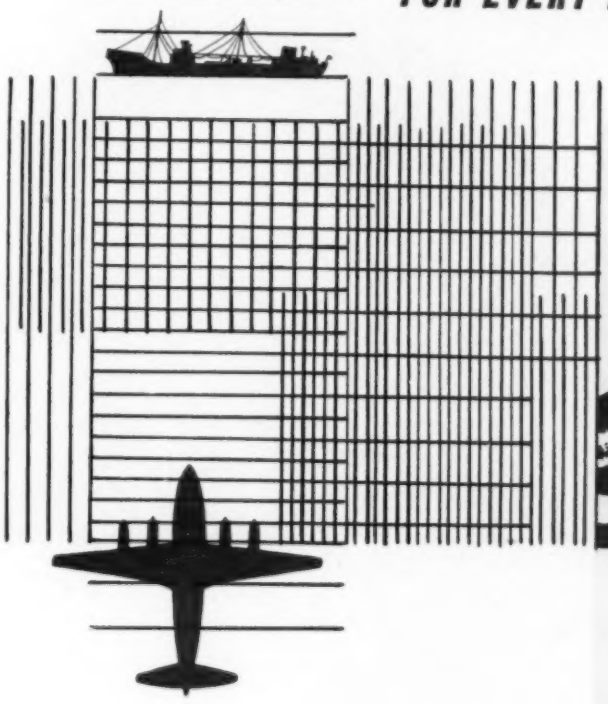

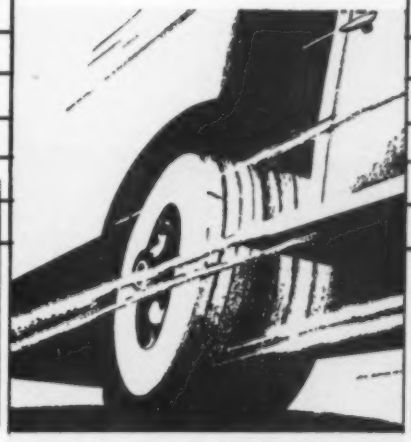

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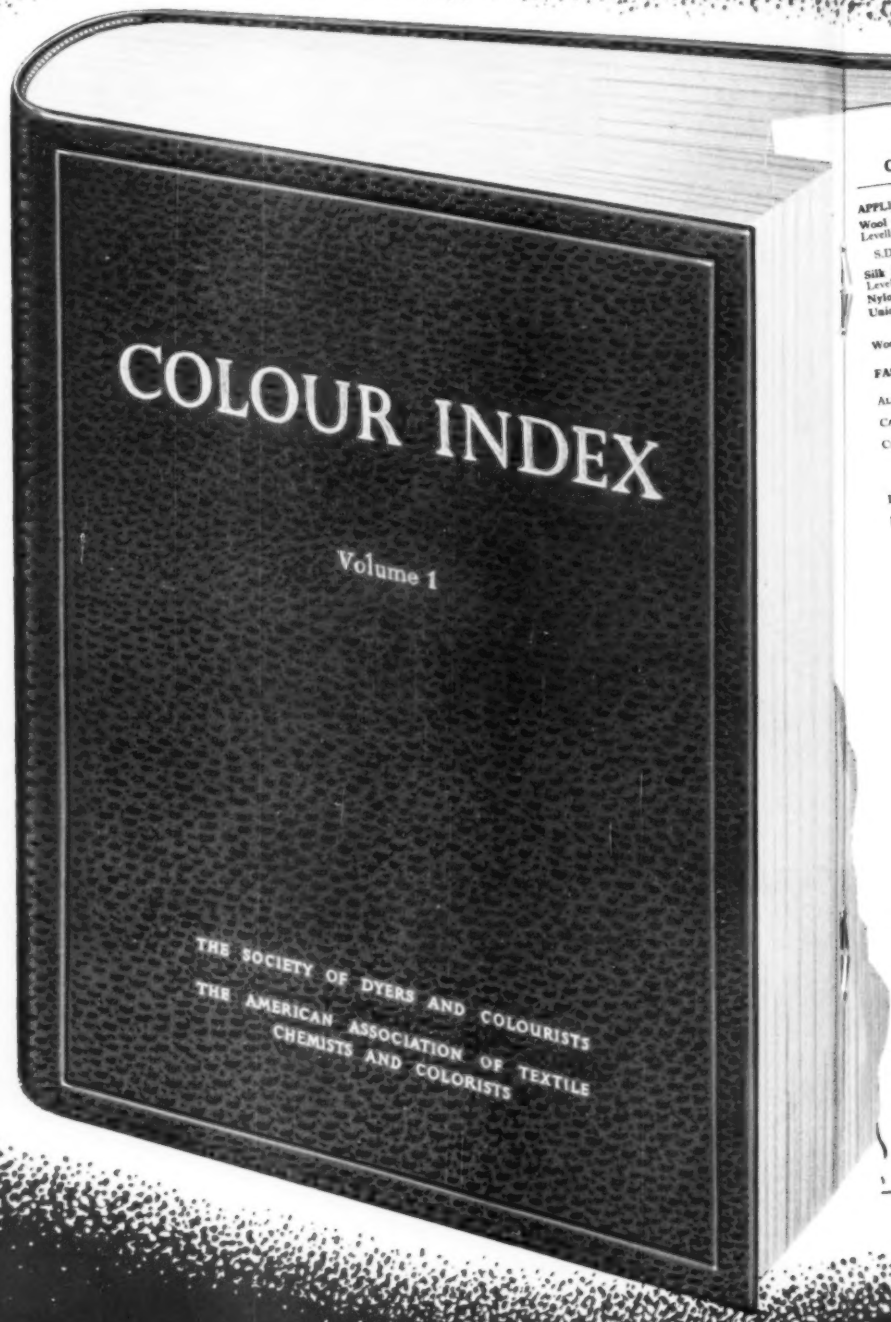

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APPLICATION DYEING

Wool Good exhaustion from a sulphuric acid dyebath
Levelling: moderate to good

S.D.C. Migration Test { Dyeing method 1 / Migration 3 } formic or sulphuric acid dyebath

Silk Dyed from a formic or sulphuric acid dyebath
Levelling: moderate to good
Nylon Dyed from a formic acid dyebath
Unions Wool dyed, nylon dyed, silk slightly stained, cellulose and acetate unstained

Method: Neutral ☐ Weak Acid ... ☐ Sulphuric Acid ■ Chrome in Dyebath ☐

PRINTING

Wool and Silk For direct print styles

FASTNESS PROPERTIES etc

	A	B	C
Alkali	5	4–5	4
Carbonising	5	4–5	4
Chlorination Alteration	5		
Chlorination Staining (Wool)	5		4
Chlorination Staining (Cotton)	5		
Decatising	4–5	4–5	4–5
Light 1/3 Normal	3	3	3
Light Normal	4	4	4
Light 2× Normal	5	4–5	4–5
Milling Alkaline Alteration			
Milling Alkaline Staining (Wool)	2	4	1–2
Milling Alkaline Staining (Cotton)		4	
Milling Acid Alteration		3	
Milling Acid Staining (Wool)		1	3–4
Milling Acid Staining (Cotton)		4–5	

	A	B	C
Peroxide Bleaching Alteration	1	5	3–4
Peroxide Bleaching Staining (Wool)		5	
Peroxide Bleaching Staining (Cotton)		5	
Perspiration Alteration	3	4–5	3
Perspiration Staining (Wool)		4–5	
Perspiration Staining (Cotton)		4–5	
Potting Alteration	5	3–4	...
Potting Staining (Wool)		2	
Potting Staining (Cotton)		5	...
Sea Water Alteration	3	2	2–3
Sea Water Staining (Wool)		3	
Sea Water Staining (Cotton)		2	
Stoving Alteration	4	4–5	4–5
Stoving Staining (Wool)		5	
Stoving Staining (Cotton)		4	
Washing Alteration	2	2	2–3
Washing Staining (Wool)		5	4
Washing Staining (Cotton)		4–5	

Dischargability Good
Solubility Water, good
Effect of Metals Copper, somewhat redder: Iron, weaker and duller

TEXTILE USAGE DYEING

Generally where high tinctorial value is of interest for the production of heavy shades of greens, browns and olive, in combination with other acid dyes. For dyeing woollen carpet yarns, light weight piece goods and hat felts
Of somewhat limited interest on silk, the fastness to water, sea water, washing, milling and preparation being a little lower than on wool

PRINTING

Generally for direct print styles on wool and silk. Dischargable to a good white on wool and silk

NON-TEXTILE USAGE

Lakes The barium lake is used for paper coating and has good fastness to light. A lake on aluminium hydroxide is used for transparent effects in tin printing
Fastness: Light, good; Heat, moderate
Leather On vegetable, chrome and semi-chrome tannages. Properties on chrome tannage (SDC): Light 4, Penetration 4
Paper For surface staining and colouring of crêped tissues
Miscellaneous For colouring milled soaps, casein plastics, anodised aluminium, writing inks and wood stains. For manufacture of light filters in photography
Used for colouring foods, drugs and cosmetics
See Food Yellow 4

Hue Yellow
Artificial Light: slightly redder

Acid Yellow AT ... YDC
Acid Yellow T ... Gy
Airedale Yellow T ... YDC
Aizen Tartrazine ... HCC
Amacid Yellow T ... AAP
Calcocid Yellow MCG, RX, XX ... CCC
Daishiki Tartrazine ... KKK
Fast Wool Yellow T ... RBM
Fenazo Yellow T ... G
Hidacid Tartrazine ... HD
Hydroxine Yellow L ... JCO
Kayaku Tartrazine ... KYK
Kiton Yellow T ... CAC
Kiton Yellow T ... Ciba
Java Yellow TA ... Vond
Lake Yellow ... NAC
Mitsui Tartrazine ... MDW
Naphtocard Yellow O ... SAC
Sen-ei Tartrazine ... KKK
Showa-Kowa Tartrazine ... KKK
Sugai Tartrazine ... LBH
Tartar Yellow FS, N, PF, S ... UIC
Tartran Yellow ... S
Tartraphenine ... JWL
Tartratol Yellow L ... Adel
Tartrazine ... Fran
Tartrazine BCC, CCC, DuP, FDN, FNC, Gy, Ipca, KO, MVC, NCC, Vond ... S
Tartrazine AF ... WSS
Tartrazine B ... Ciba
Tartrazine (*Biological stain*) ... CCC
Tartrazine C ... G
Tartrazine J ... Acna
Tartrazine Lake Yellow N ... ICI
Tartrazine M ... ICS
Tartrazine N ... ICI
Tartrazine NS ... NSK
Tartrazine O ... FH
Tartrazine XXX ... PbC
Tartrazol BPC ... W
Tartrazol Yellow ... W
Tartrine Yellow TE ... BEL
Unitertracid Yellow O ... CT
Wool Yellow ... NAC

C.I. 10315 Nitro

Dyes wool and silk from a weak acid dyebath but rarely used in normal dyeing. Used as a mothproofing agent for wool felts. Limited use in the colouring of varnishes

Hue Yellow

Manchester Yellow ... WSS
Martius Yellow ... Fran
Pure Golden Yellow ... VIt

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THE NEW COLOUR INDEX

Produced by

THE SOCIETY OF DYERS AND COLOURISTS

THE AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

The work has been completely re-designed and brought up-to-date with the co-operation of all the dye and pigment manufacturers in Great Britain, America, Western Europe, India and Japan.

It is in three main parts

PART I consists of two volumes, containing more than 1,700 pages in all. It deals with all commercially homogeneous dyes and pigments in current use (approximately 4–5,000 dye entities and 25,000 commercial names) within groups relating to their main usage, e.g. Disperse, Acid, Azoic, Direct, Pigment, Solvent etc. The data included comprises the commercial name and manufacturer's code letters, methods of application, fastness properties, established usages, reactions on the fibre and literature references relating to application. In the absence of a single accepted international system of fastness assessment at the time of compilation (although there is *now* an accepted international system) it was necessary in this Edition to make provision for the inclusion of data according to one or more of the following —

A — (American)	The methods of The American Association of Textile Chemists and Colorists
B — (British)	The methods of The Society of Dyers and Colourists
C — (Continental)	The methods of The German Fastness Committee

An important feature of Part I is the sub-division of each usage group into hue groups — Yellow, Orange, Red, Violet, Blue, Green, Brown and Black. The hue classification has been made by reference to a Hue Indication Chart which has been designed specially for the purpose. A copy of the chart is included with each set of volumes.

PART II is one volume of over 800 pages. It contains data relating to chemical constitution, method of preparation, patent and literature references relating to manufacture or constitution, solubilities and reactions in substance for approximately 3,800 dyes and pigments. The 1st Edition recorded 1,316 constitutions, and this very big increase is partly due to the fact that many co-operating dye manufacturers have, for the first time, disclosed the constitution of certain of their dyes. In addition there is the information contained in the reports compiled by Allied Investigating Commissions on the German Dye Industry.

The dyes are arranged in order based on their chemical constitution following broadly the lines of the 1st Edition. A number of refinements have been introduced and there are two new sections, viz. Azoic and Phthalocyanine.

Each entry carries a five digit reference number. These numbers do not progress in unit steps as provision has been made to allow the insertion of new constitutions in later editions or supplements without having to change the numbers now allocated in the 2nd Edition. Each entry also carries the complementary Part I designation thus providing easy cross reference to the data in Part I. It also contains alphabetical and empirical formula indexes to all the Intermediates relating to individual dye entities.

PART III is one volume of over 500 pages containing the general index to Part I and Part II. The Commercial Names Index includes the names (in italics) of dyes not in current use, many of which are related to data in Part I and or Part II.

The price is £40 per set of 4 volumes, plus carriage on overseas consignments, and payment in advance of delivery is required

Full particulars from

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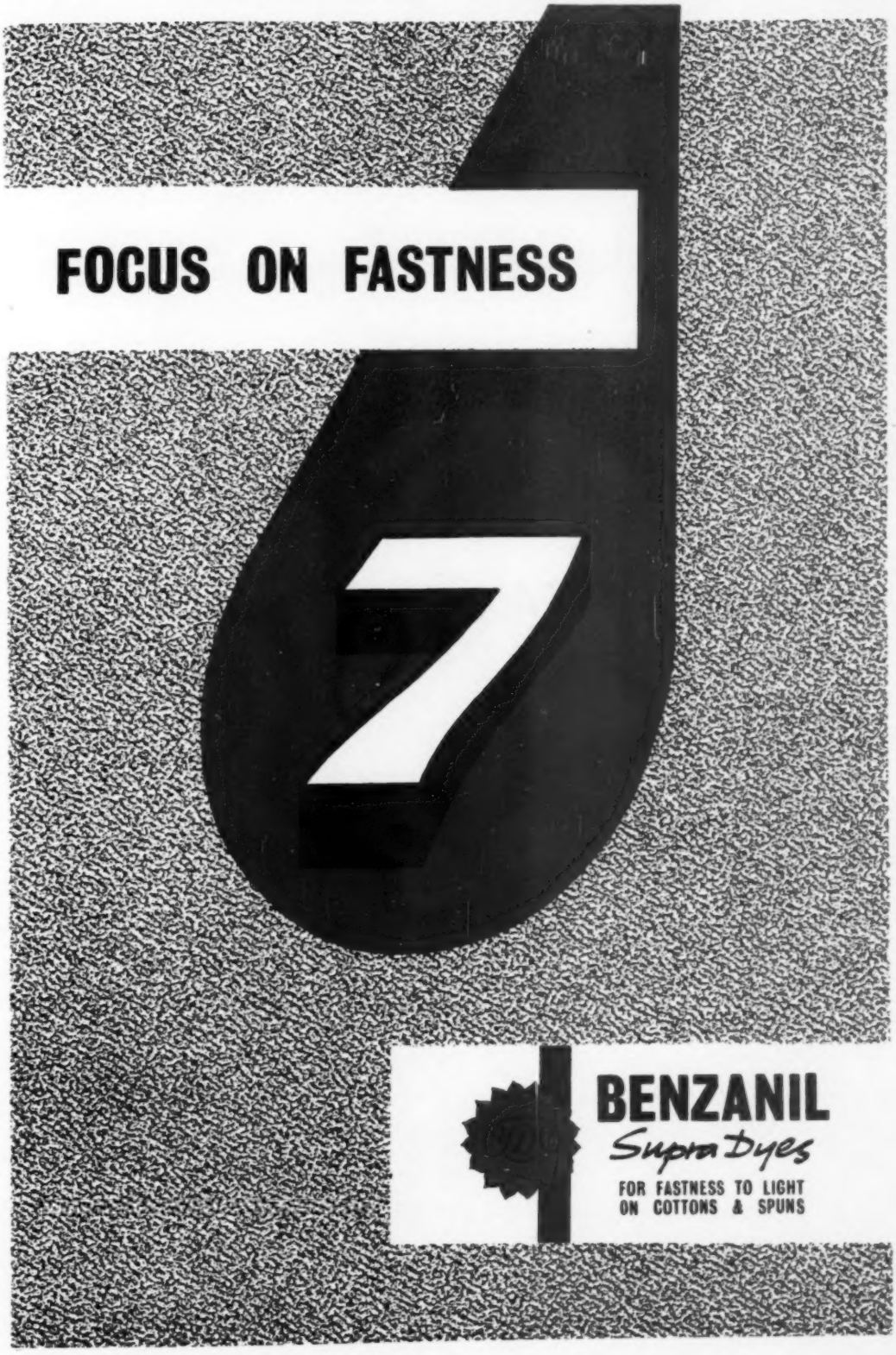

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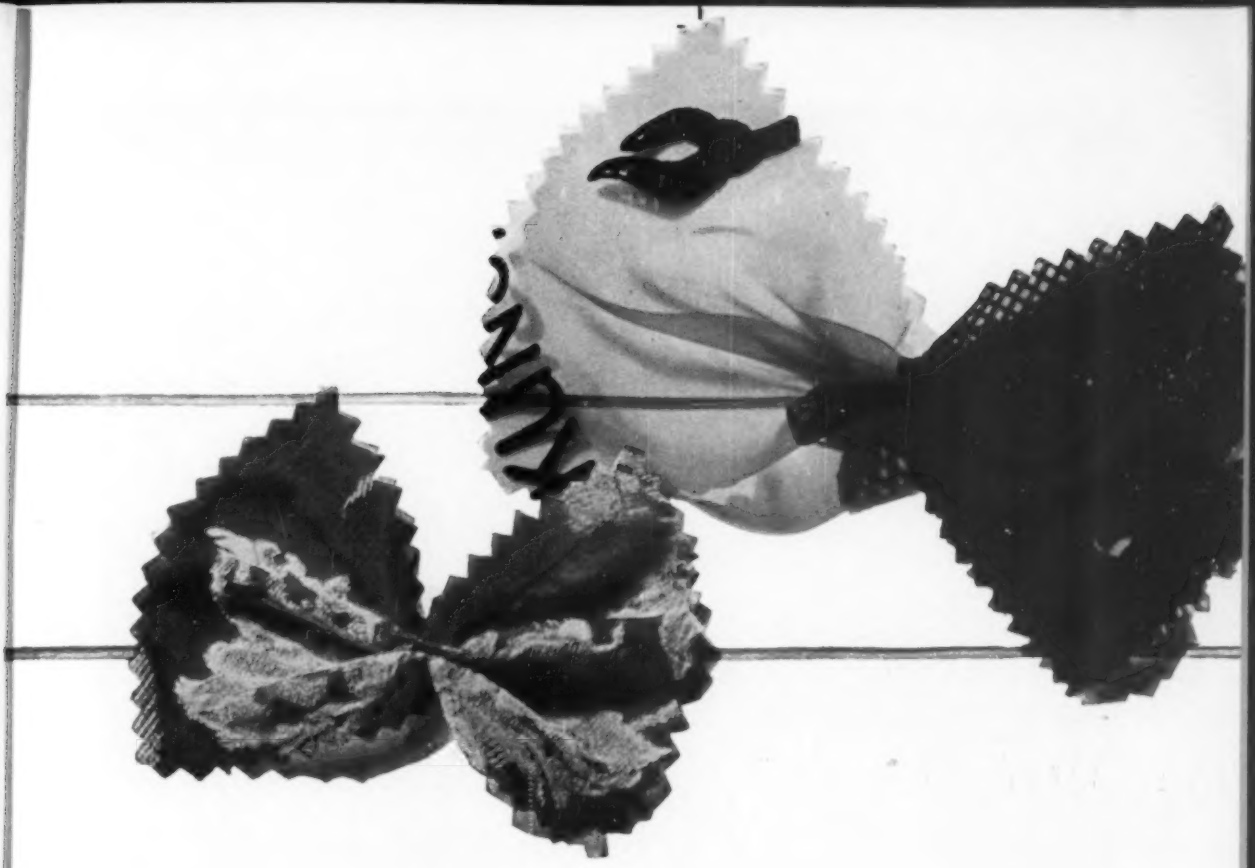

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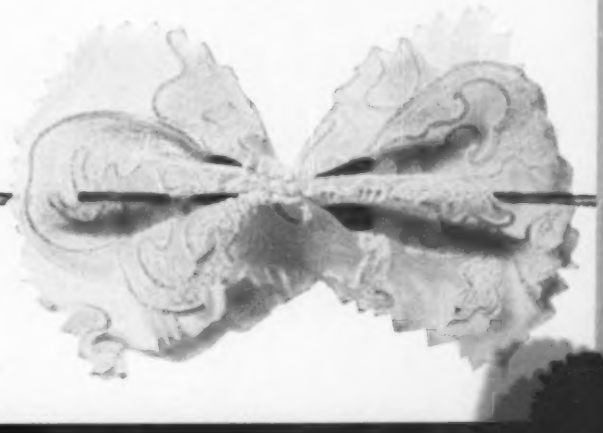

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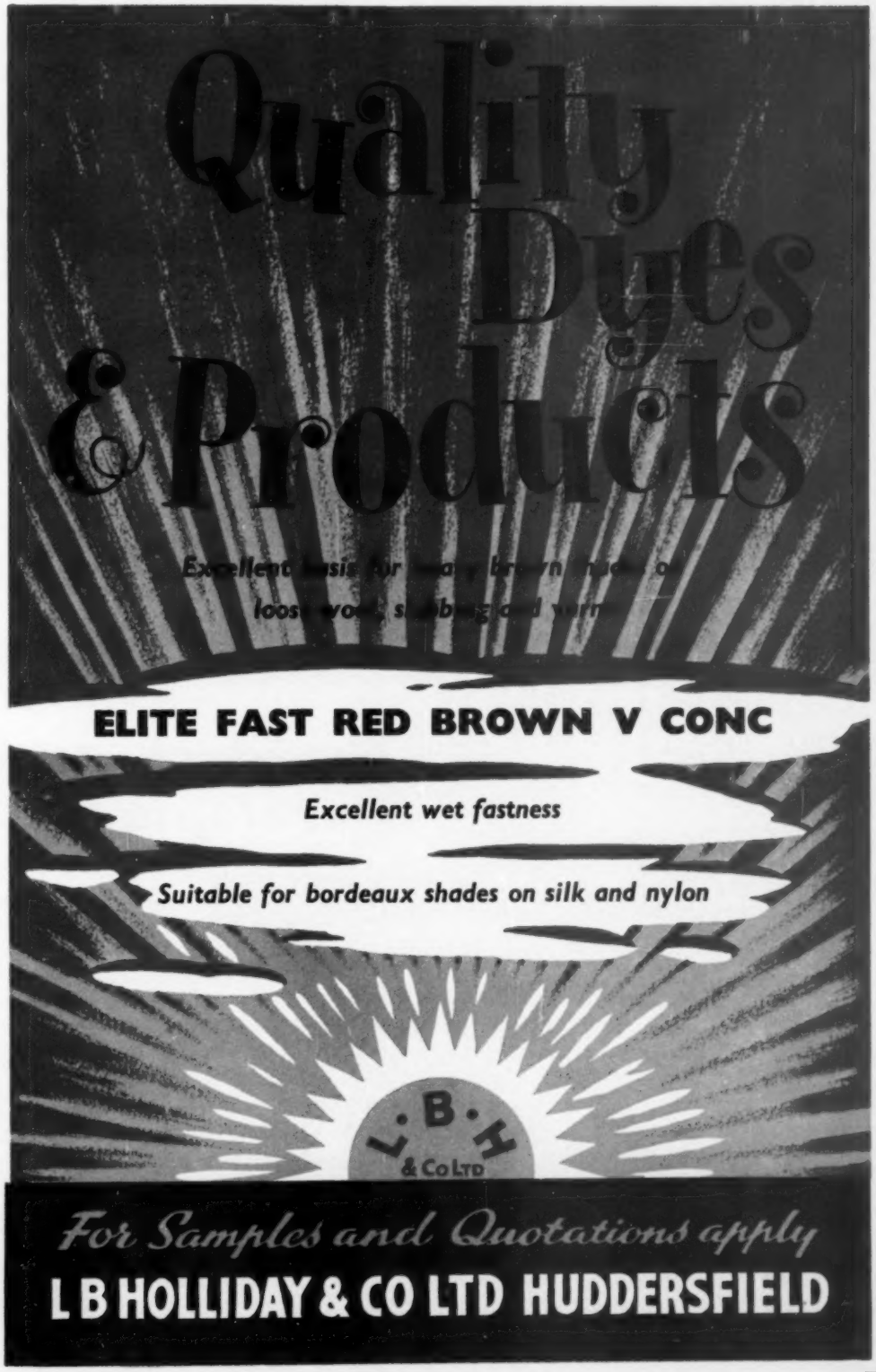
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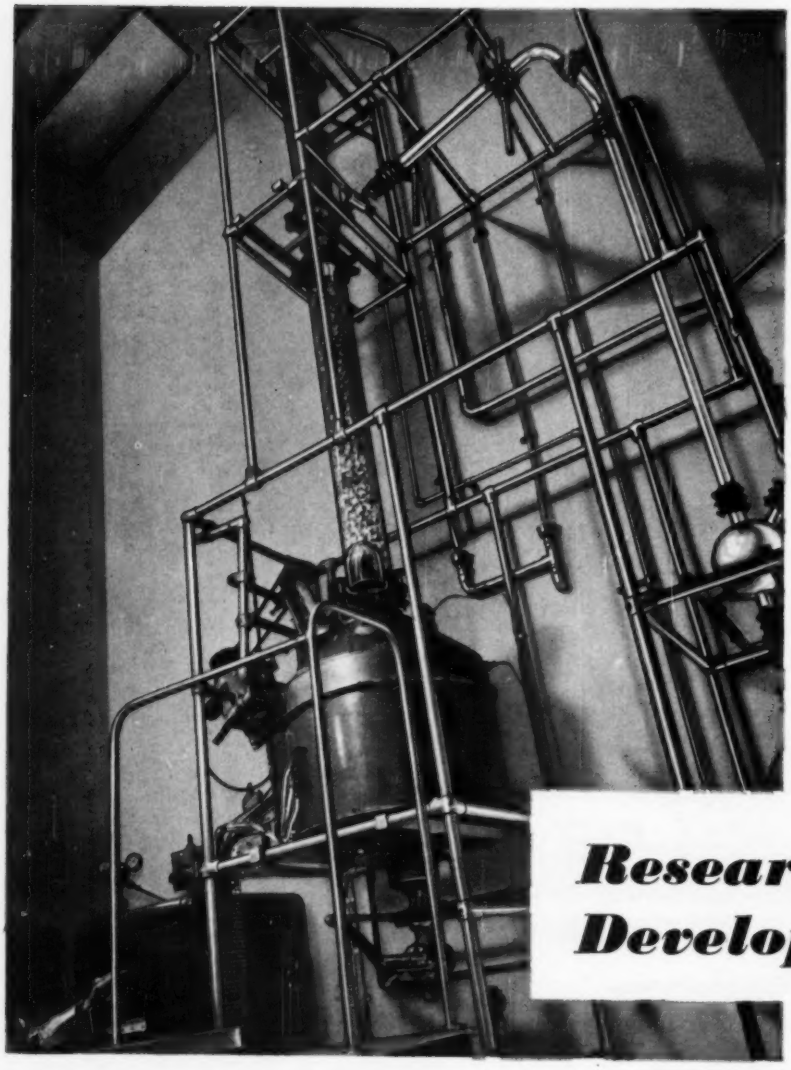

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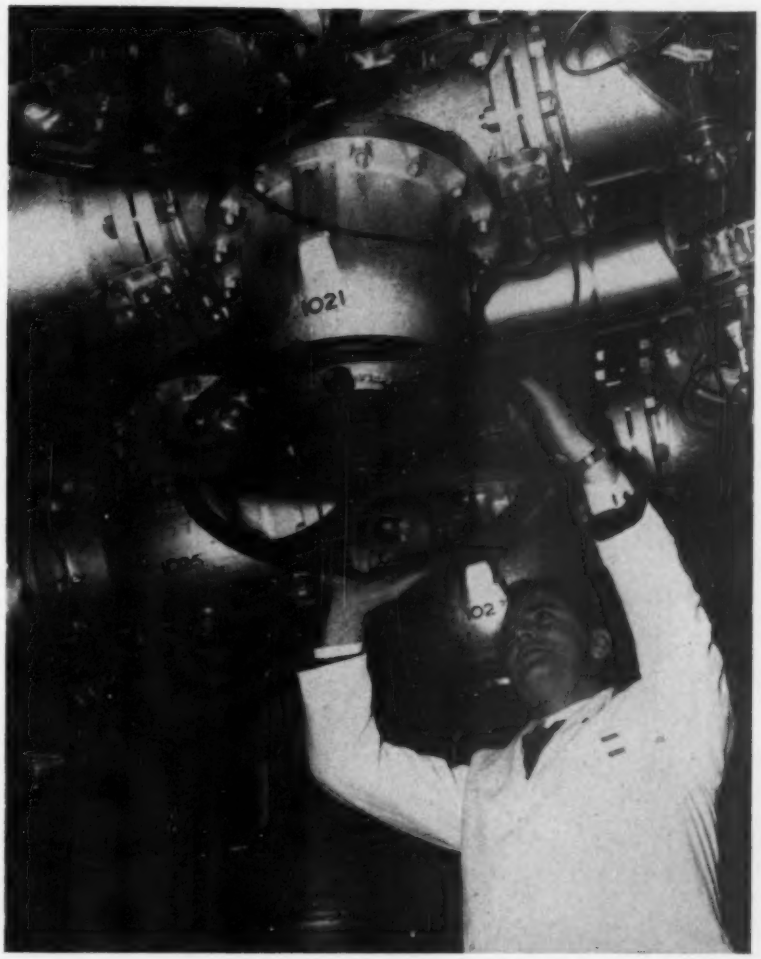

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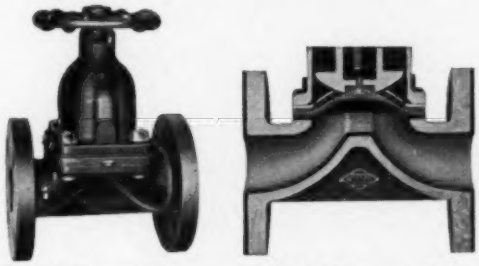

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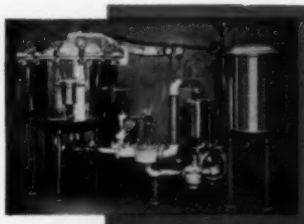
A complete range of machines for dyeing all types of fibre in every stage of manufacture, including loose, top, beam, hank and package forms. Also rapid package drying machines.



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Built in stainless steel in capacities ranging from 25 to 1500 lbs. for all classes of wool, worsted, mixture and linen yarns in hank form. Machines may be coupled together.

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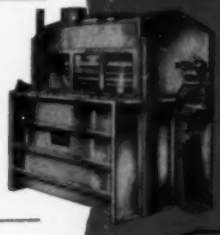

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Totally enclosed rotating drum machine for dyeing nylon, pure silk, rayon and cotton hose with perfect penetration and uniformity. Stainless steel in 25, 50, 100, and 200 lbs. capacities.

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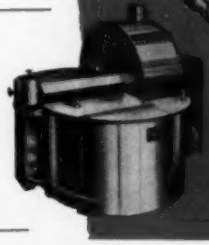
A versatile machine, in stainless steel, for ladies' hose, half hose, and loose articles. Also used for anti-shrink and bleaching. 4 capacities from 12–200 lbs.



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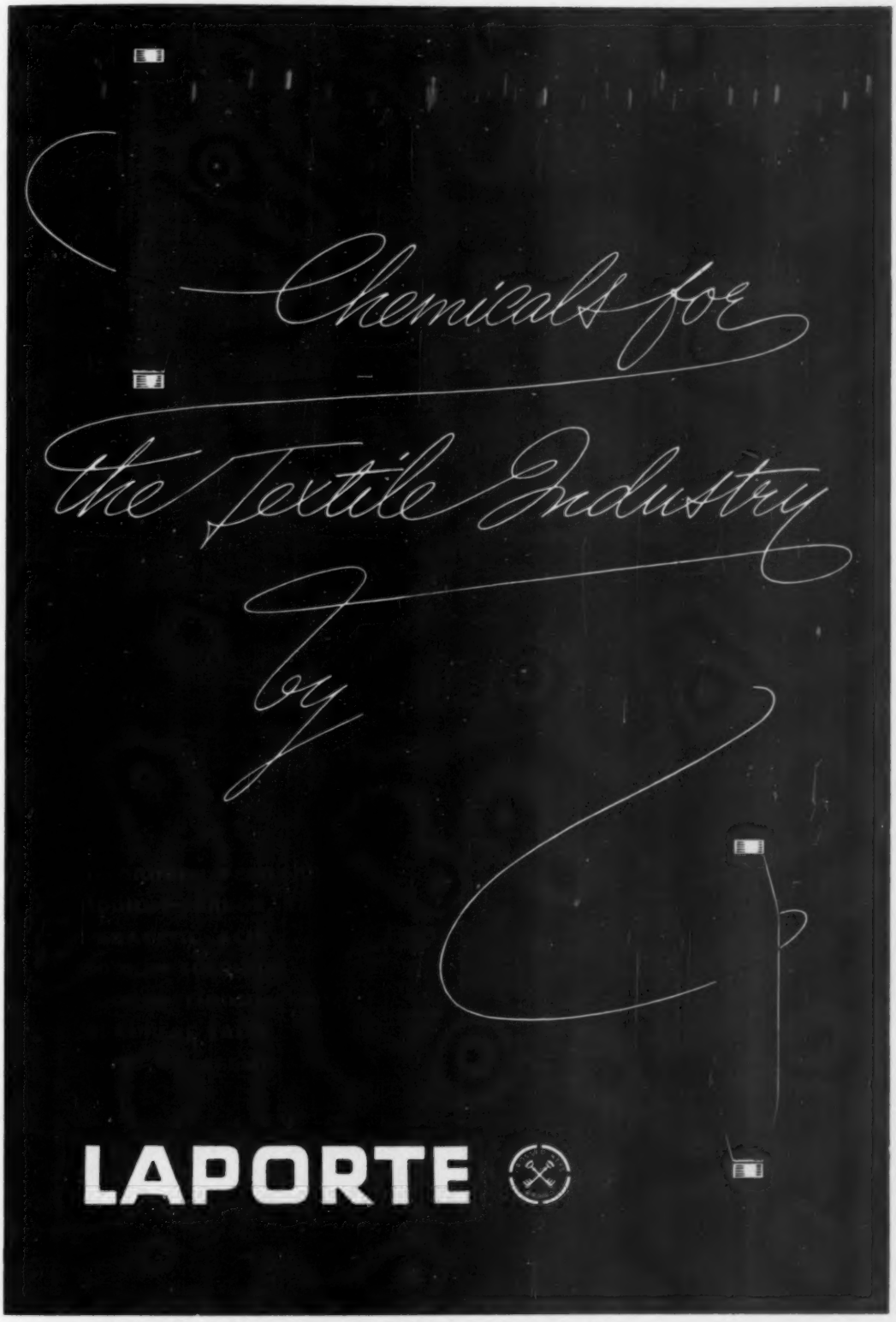

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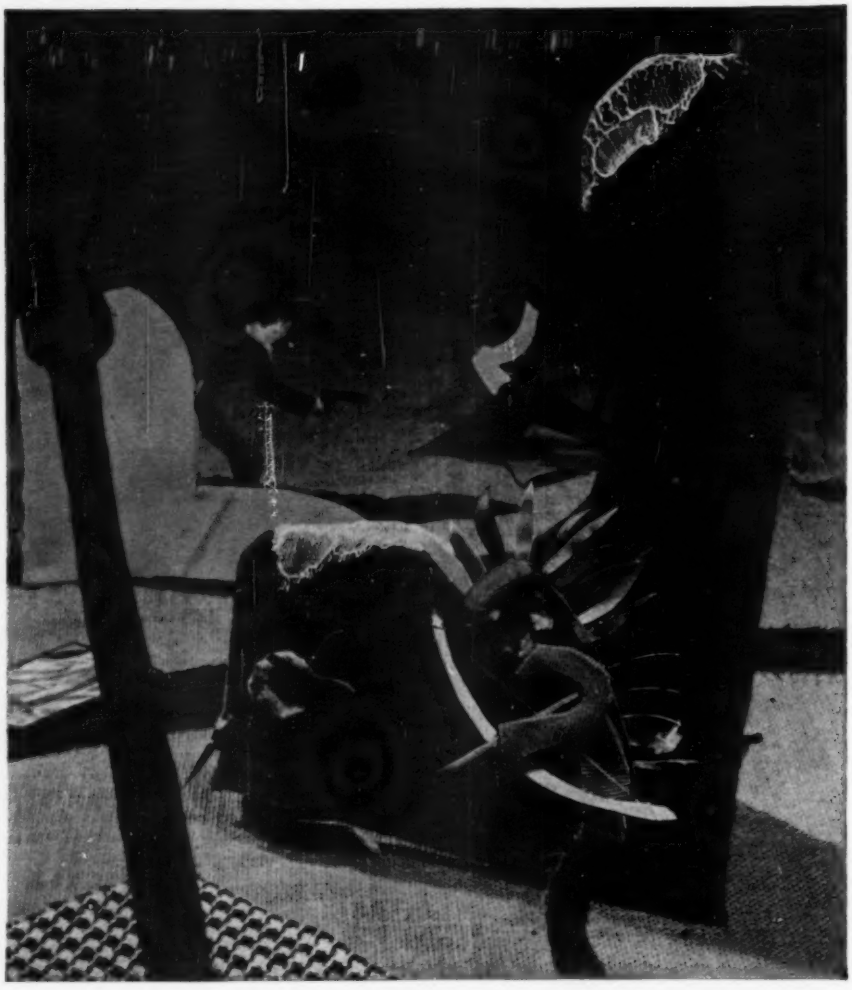

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The Maxilon dyestuffs are designed specifically for application to polyacrylonitrile fibres to give excellent build up and fastness properties.

200 years Geigy

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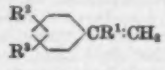
A 53.

Size for Nylon
Monsanto *USP* 2,808,348
An aqueous solution of a terpolymer of vinyl acetate, maleic anhydride and an alkoxyethyl partial ester of maleic acid is an excellent size for nylon, from which it can be removed by simple scouring. C.O.C.

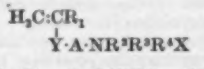
Acidic Sizes
Monsanto *BP* 799,758
Terpolymers of vinyl acetate, maleic anhydride and an olefinic substance are stable products useful as sizes for nylon and other yarns requiring an acidic size. The ratio of vinyl acetate to maleic anhydride is 52–48 to 50–50 mol. %. The olefinic substance is either an alkyl acrylate or methacrylate (Alk of 1–8 C) and forms 1–30 mol. % of the terpolymer or is a compound of formula—



(R^1, R^2 and R^3 = H, Hal or Alk of 1–4 C) and forms 1·8–4·0 mol. % of the terpolymer. C.O.C.

Antistatic Lubricant for Organic Derivatives of Cellulose
Celanese Corpn. of America *USP* 2,805,992
A composition composed of mineral oil, a bodied sperm oil, a higher fatty phosphate, a tertiary amine and a non-ionic surface active agent is very stable, imparts excellent lubrication and antistatic properties to cellulose acetate and is readily removed by scouring in water. C.O.C.

Antistatic Agents
Rohm & Haas Co. *USP* 2,808,349
Compounds of formula—



(R^1 = H or CH_3; Y = O, $CONR^1$ or COO; A = straight or branched chain alkylene of 2–10 C which may contain 1 or more hydroxyl groups, at least 2 of the C atoms are connected in a chain between N and Y; R^2 and R^3 = separately, saturated or ethylenically unsaturated hydrocarbons of 1–4 C, or together form a morpholino, pyrrolino, piperidino or *N*-alkylpiperazino > $(C_2H_4)_2$: NR^4 (R^4 = Alk of 1–18 C); R^4 = allyl or methallyl; X = negative salt-forming atom or radical), are used as antistatic agents. Thus a 5% aqueous solution of an alkylated homopolymer of dimethylaminoethyl vinyl ether applied to polythene gives an antistatic finish of good fastness to washing. C.O.C.

Antistatic and Easy-wetting Finish
Dexter Chemical Corpn. *USP* 2,809,159
An antistatic finish contains a hydroxyamine containing a large proportion of hydrocarbons and a polyhydric alcohol, e.g. an amine derivative containing 15–60 alkylene oxide units and a polyglycol of mol. wt. 200. Material treated with such a composition is readily wetted, e.g. nylon so treated is as readily wetted as cotton. C.O.C.

Textile Lubricant
National Lead Co. *USP* 2,805,993
A spinning lubricant suitable for any type of fibre consists of an aqueous emulsion of a textile lubricating oil together with 5–10% on the weight of the oil of an organophilic bentonite capable of dispersing to form a gel in organic liquids, e.g. Bentone 18 (a sodium type bentonite treated with octadecyl ammonium halide). C.O.C.

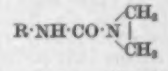
Antistatic Agents for Use on Hydrophobic Man-made Fibres
Pennsalt Chemical Corpn. *USP* 2,805,964
Sulphones, particularly those of formula $(R^1SO_2R^2)_xR^3$ (R^1 = Alk of 3–6 C; R^2 = divalent hydrocarbon of 1–6 C; R^3 = OH which may be esterified with a saturated aliphatic monocarboxylic acid of 1–17 C, a saturated aliphatic dicarboxylic acid of 2–10 C or a benzene dicarboxylic acid in which the COOH groups are attached to the ring; x = 1 when R^3 = OH and to the numbers of COOH groups in the acid from which R^3 is derived when R^3 is an ester radical) e.g. 2-hydroxyethyl sulphone or 2-pelargonoxyethyl sulphone, are excellent antistatic agents for polyacrylo nitriles, polyester, and similar man-made fibres. C.O.C.

Detergent
Eastman Kodak Co. *USP* 2,805,205
Addition of copolymers of acrylonitrile and acrylamide or an alkyl acrylamide to anionic or nonionic detergents prevent redeposition of soil on textiles when they are washed with these detergents. C.O.C.

Water-soluble Biguanide Condensates for Improving the Dyeing Properties of Cellulosic and Polyamide Fibres
ICI *BP* 798,061
Water-soluble, viscous bases are obtained by heating a mixture of a compound of formula $A[NXC(NH)\cdot NH\cdot C(NH\cdot NH_2)]p$ (A = H, Alk, alkylene, Ar or arylene; p = 1 or 2; X = H or Alk) and one of formula $Y[(CH_2)_nNH]_m(CH_2)_nNH_2$ (Y = NH_2; n = 2 or 3; m = > 1), e.g. a mixture of biguanide and triethylenetetramine, under anhydrous conditions. Cellulose treated with an aqueous solution of such a base at 50–100°C. can be dyed with acid and mordant dyes, polyamide fibres so treated absorb several times more dyes that are solubilised by anionic groups than do the untreated fibres. The bases can also be used for improving the wet fastness properties of direct dyeing on cellulose. C.O.C.

Acetylene Diurea–Formaldehyde Resins for Finishing Textiles
Dan River Mills *USP* 2,803,564
Good powers of recovery from creasing and dimensional stability are obtained by the use of condensates of 1 mol. tetramethylol acetylene diurea and 6–17, preferably 9 mol. of formaldehyde for the resin finishing of textiles. The resultant finish has no tendency to retain chlorine when treated with bleaching agents containing chlorine. C.O.C.

Stable Finishing Compositions containing *NN*-Alkyleneurea Compounds
FH *BP* 799,045
Composition containing compounds of formula—



(R = an aliphatic or homocyclic radical of > 9 C) e.g. *N*-dodecyl-*N'N'*-ethylene urea, urea and a water-soluble inorganic salt which can exist with water of crystallisation but which is not deliquescent in any form, e.g. anhydrous Na_2SO_4, are stable even on prolonged storage at 40°C. C.O.C.

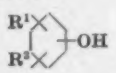
Lithium Thioglycollate as Cold Waving Composition
Asbo Laboratories *BP* 798,674
Lithium thioglycollate is at least as active as NH_4 thioglycollate and under most conditions is even more active. It has the advantage of being odourless. C.O.C.

Solvents or Swelling Agents for Polyethylene Terephthalate
FH *BP* 797,425
Compounds of formula $X^1C_nH_{2n}X^2$ (X^1 = COOH, NO_2, CH_2Cl, $CHCl_2$, CCl_3 or COOR (R = CH_3 or C_2H_5); X^2 = OH, CR:O, COOH or COOR; n = 1–4 and may also be O in the case of halogeno groups; the C chain of the C_nH_{2n} group may be interrupted by O), e.g. a mixture of chloroacetic acid (40 parts by wt.) and trichloroacetic acid (60), are solvents for polyethylene terephthalate. C.O.C.

Polyvinyl Chloride Pastes for Coating Textiles
Chemische Werke Huls *BP* 799,953
Addition to pastes of polyvinyl chloride and softening agents of up to 1% (on wt. of the polyvinyl chloride) of a silicic acid obtained by burning a mixture of $SiCl_4$ and H makes them applicable to textiles without there being any penetration into the textiles. Textiles coated with such pastes have good handle after gelatinisation. C.O.C.

Tanning Agents
FBy *BP* 799,224
Modification of *BP* 747,675 (J.S.D.C., **72**, 358 (1956)) Tanning agents having improved dispersing properties are prepared by adding a lower saturated aldehyde, e.g. HCHO, and an *o*-chlorosubstituted phenol having at least one free *o*- or *p*-fraction, e.g. 6-chloro-*m*-cresol, simultaneously but separately into a mono- or disulphonic acid of an aromatic hydrocarbon containing at least two

condensed aromatic nuclei, e.g. β-naphalene sulphonic acid. A phenol of formula—



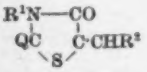
(R^1 = Alk, Hal, NO_2 or carbalkoxy; R^2 = H, Alk, Hal, NO_2 or carbalkoxy or $R^1 + R^2$ = a benzene nucleus) e.g. *p*-chloro phenol, is either incorporated into the acid before the process starts or is added together with the *o*-chloro substituted phenol. C.O.C.

Yellow Colloidal Silver for Absorbing Blue Light
DuP *USP* 2,806,798

Yellow colloidal silver sols of extremely fine particle size, excellent blue-light absorption and no significant fogging or desensitising action on silver halide emulsions are obtained by reducing a AgBr sol. in presence of a protective colloid with a reducing agent of reduction potential $\not< 0{\cdot}3$, e.g. hydrazine hydrate. C.O.C.

Ultraviolet-absorbing Agents for Use in Colour Photography
Eastman Kodak Co. *USP* 2,808,330

Compounds of formula—



(R^1 = H, Alk or Ar; R^2 = Ar; Q = divalent, non-metallic atom or radical), e.g. 5-benzal-3-ethylrhodanine or 5-benzal-3-methyl-2:4-thiazolidine, are excellent ultraviolet absorbing agents for use in colour photography. C.O.C.

Colour Reaction for Non-ionic and Amphoteric Soaps (VIII p. 718)
Triazine Derivatives as Paper Sizes (XI p. 724)
Laboratory Screening Test for Bactericides for Use in Sheepskin Soaking Pits (XII p. 725)
Resins as Tanning Agents (XII p. 726)
Polyalkylene Oxide Wax Derivatives for Oiling Fur Skins (XII p. 726)

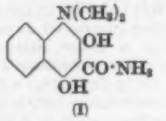
IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Oxidation of Naphthalene by Air in the presence of Vanadium Catalysts in Gaseous Suspension
Z. Gropşian and I. Krispin
Studii şi Cercetări Ştiinţifice, Timişoara, Chimie, 1, 79–82 (Jan.–Dec. 1954)

The efficiency of a vanadium catalyst supported on silica gel for the air oxidation of naphthalene is found to be almost double, with respect to the yield of phthalic anhydride, that afforded by fixed vanadium catalysts on different supports. Moreover, the reaction temperature with the former catalyst is lower, the resulting product is purer, and the erosion of the plant is reduced. H.H.H.

4-Substituted 1:3-Dihydroxy-2-naphthoic Acid
H. Bretschneider and H. Rogenhofer
Mh. Chem., 88, 652–662 (Sept. 1957)

For the testing of antibacterial activity, 4-dimethylamino-1:3-dihydroxy-2-naphthoamide (I) has been synthesised by the following independent routes—(1) 4-bromination of ethyl 1:3-dihydroxy-2-naphthoate followed by replacement of the Br by reaction with dimethylamine and subsequent aminolysis to I; and (2) owing to the difficulty of the last named stage, the starting substance was 1:3-dihydroxy-2-naphthoamide, which brominated in the 4-position and on subsequent treatment with dimethylamine afforded I.



(I)

H.H.H.

Separation of the 1:5- and 1:8-Naphthylaminesulphonic Acids (5- and 8-Aminonaphthalene-1-sulphonic Acids)
J. Reichel, P. Iacobescu, and I. Penescu
Studii şi Cercetări Ştiinţifice, Timişoara, Chimie, 1, 43–46 (Jan.–Dec. 1954)

Two methods of separation of the title acids from technical mixtures are compared, viz. that of salting out and that based on the different basicities of the isomers. Both have approximately equal efficiencies but the latter is simpler although more expensive. H.H.H.

Internal Complex Salts of Azo Compounds. V—Simultaneous Dealkylation and Chelation of Copper with *o*-Hydroxy-*o'*-alkoxyazo Compounds
V. I. Mur
J. Gen. Chem. U.S.S.R., 28, 998–1002 (April 1958)

Dealkylation of *o*-hydroxy-*o'*-methoxyazo compounds with equivalent amounts of $CuSO_4$ and pyridine gives a 1:1 complex. On the other hand, dealkylation of such a compound to give a 1:2 complex is more difficult. The effect of substituents in the benzene nucleus, in the *para* position to the methoxyl group, is investigated, and ease of dealkylation occurs in the following order: $NO_2 > H > O{\cdot}CH_3 > CH_3 > Cl$. However, the presence of a nitro group in the *meta* position to the methoxyl group renders dealkylation more difficult. An explanation of the action of $CuSO_4$, in the presence of an organic base, on such alkoxyazo compounds is given, and a mechanism suggested. G.J.K.

Metal-complex Salts of Dyes
Z. Yoshida
Yûki Gôsei Kagaku Kyôkai Shi, 16, 240–251 (1958): *Chem. Abs.*, 52, 11427 (10 July 1958)

A review of dyes of the quinone oxime, *o*-hydroxyquinone, *o*-hydroxycarboxylic acid, phthalocyanine, azomethin, and 8-quinolinol series, and azo dyes having a coordinating atom in the *ortho* position. 197 references. C.O.C.

Steric Effects on Mesomerism

XVII—Some Properties of Aromatic Nitro, Amino, and Acylamino Compounds with Bulky ortho-Substituents
J. Burgers, M. A. Hoefnagel, P. E. Verkade, H. Visser, and B. M. Wepster
Rec. Trav. chim., 77, 491–530 (June 1958)

The influence is discussed of 2-methyl-6-*tert.*-butyl (I) substitution and/or 2:6-di-*tert.*-butyl substitution (II) on certain properties of cpd. such as nitrobenzene, aniline, *N*-methylaniline, *NN*-dimethylaniline, acetanilide, 4-nitroaniline, and 4-nitroacetanilide. For deriv. of nitrobenzene, *NN*-dimethylaniline, and acetanilide, the electronic absorption spectra indicate a complete or almost complete elimination of the mesomeric interaction, but for all the primary amines investigated the mesomerism is fully developed, while the secondary amines take up an intermediate position. Abnormally lower basic strengths of I-substituted primary amines and II-substituted *NN*-dimethylanilines are ascribed to steric hindrance to solvation. The basic strength of 3:5-di-*tert.*-butyl-4-nitroaniline affords evidence for the quant. significance of the inductive and mesomeric effects of the nitro group in 4-nitroaniline as well as on the *meta*/*para* ratio of the inductive effect. Some reaction rate constants relating to catalytic deacylation are discussed.

XVIII—Steric Inhibition of the Mesomerism in Aromatic Amines with Bulky *N*-Alkyl Groups of Compact Structure—Nitrosation and Coupling of the four *N*-Butyl-*N*-methylanilines
T. C. van Hoek, P. E. Verkade, and B. M. Wepster
Ibid., 559–568

Nitrosation and coupling experiments with the four *N*-butyl-*N*-methylanilines are described, of which the *N*-*tert.*-butyl-*N*-methylaniline could not be nitrosated (thereby confirming the experience of Hickinbottom, *J.C.S.*, 946 (1933)) and reacted only very slowly with actively coupling diazonium salts. H.H.H.

Reduction Products obtained from Nitrogen Compounds under the Action of Ramey Nickel and Hydrazine Hydrate
S. Hornsby and W. L. Peacock
Chem. and Ind., 858–859 (5 July 1958)

The following extensions are reported to the known

range of N cpd. which may be reduced by hydrazine hydrate (I) soln. in presence of Raney nickel catalyst (II)— azobenzene (III) to hydrazobenzene (IV), azoxybenzene to III and to IV, phenylhydroxylamine to IV, nitrosobenzene to IV and also to aniline (V), and benzylideneaniline to V (with elimination of benzaldehyde) together with a small amount of a suspected secondary amine. The method employed was to treat the organic N cpd. (1 g.) in absolute alcohol (10 c.c.) with I (2–3 mol.; 100%), heat the mixture until homogeneous, add 0·1 g. of II (10% in alcohol), reflux until the colour is discharged, then destroy excess I with more of II, filter off the nickel, and work up the solution for individual cases as described. H.H.H.

Direct Introduction of the Diazonium Group into Aromatic Nuclei. IV—Preparation of Diazonium Salts from Phenols
J. M. Tedder and G. Theaker
J.C.S., 2573–2579 (July 1958)

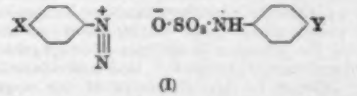
A general method is reported for the preparation of diazonium salts from phenols based on reaction with buffered nitrous acid in aq. acetone, which proceeds *via* the nitrosophenol and shows all the usual characteristics of electrophilic substitution. The reaction stops at the nitrosophenol (quinone monoxime) stage, however, when the parent quinone is particularly stable, as in the naphthols. Phenols, when treated with HCl and an alkyl nitrite dissolved in the corresponding alcohol, yield diazonium salts derived from the related alkyl phenyl ether. H.H.H.

Interaction of Cyclic Salts of Salicylic Acid with Nitrous Acid. Preparation of 5-Diazosalicylic Acid
A. A. Nemodruk
J. Gen. Chem. U.S.S.R., **28**, 1082–1085 (April 1958)

Al, Cr, Zn, Be chelates of salicylic acid react in acid solution with nitrous acid giving 5-diazosalicylic acid, 5-nitrososalicylic acid being an intermediate product. In contrast to salicylic acid itself, the cyclic salts prevent the elimination of CO_2 and also enhance reactivity towards nitrous acid. G.J.K.

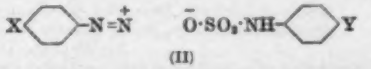
Reaction of Diazo Compounds with Sulphamic Acid and its Derivatives. IX—Causes of the Colour of Diazonium Salts of Arylsulphamic Acids
D. Z. Zavel'skiĭ and L. A. Lishnevskaya
J. Gen. Chem. U.S.S.R., **28**, 1010–1019 (April 1958)

Diazonium salts of sulphamic, benzylsulphamic, and *N*-ethylphenylsulphamic acids have been prepared. The authors claim that I is the formula for the colourless, or only weakly coloured, diazonium salt—



(I)

The deeply coloured compounds are claimed to possess a "special type" of salt-linkage which allows electron transmission (conjugation) between the two entities, and such a structure is formulated—



(II)

The greater the electron-attracting power of X, and the greater the electron-donating effect of Y, the more is formula II favoured. G.J.K.

Chemical Effect of Light on Sodium *p*-Methoxybenzenediazosulphonate
R. Dijkstra and J. de Jonge
Rec. Trav. chim., **77**, 538–554 (June 1958)

Kinetic measurements indicate that ultraviolet irradiation of an aq. soln. of *p*-methoxybenzenediazosulphonate (I) causes dissociation of the sulphonate ion into a *p*-methoxybenzenediazonium ion and a sulphite ion, which recombine in the dark to form the original sulphonate ion according to the scheme—

$$CH_3{\cdot}O{\cdot}C_6H_4{\cdot}N{:}N{\cdot}SO_3^- \underset{\text{(Light)}}{\overset{\text{(Dark)}}{\rightleftharpoons}} CH_3{\cdot}O{\cdot}C_6H_4{\cdot}N_2^+ + SO_3^{2-}$$

The occurrence of a labile or *cis*-diazosulphonate can practically be excluded. In the preparation of I the occurrence of such a labile form was never observed. H.H.H.

Mechanism of Diazotisation and Mode of Action of Carcinogenic Substances
H. Schmid
Mh. Chem., **88**, 161–164 (Feb. 1957)

The nitrogen atom is shown to be the active centre in the carcinogenic aromatic amines, and an explanation based on the author's proposed mechanism of diazotisation (*ibid.*, **87**, 560 (1956) and numerous previous papers) is suggested for the carcinogenic activity of *β*-naphthylamine and 2-aminoanthracene and for the absence of such activity in *α*-naphthylamine and 9-aminoanthracene. H.H.H.

Kinetics and Mechanism of Diazotisation
XI—Kinetics of Diazotisation in Nitrite Solutions
H. Schmid and A. Woppmann
Mh. Chem., **88**, 411–414 (June 1957))

On the basis of recorded kinetic data, the general reaction scheme of diazotisation is found to be as follows—

(1) $R{\cdot}NH_3^+ \rightleftharpoons R{\cdot}NH_2 + H^+$

(2) $HNO_2 \rightleftharpoons NO_2^- + H^+$

(3) $HNO_2 + H^+ \rightleftharpoons H_2O{,}NO^+$

(4) $H_2O{,}NO^+ + NO_2^- \rightleftharpoons N_2O_3 + H_2O$

(5) $H_2O{,}NO^+ +$ Nitrosyl-ion acceptor $(Cl^-, Br^-, HClO_4, HNO_3) \rightleftharpoons$ Nitrosyl cpd. $+ H_2O$

(6) $H_2O{,}NO^+ + R{\cdot}NH_2 \rightarrow R{\cdot}NH_2{\cdot}NO^+ + H_2O$

(7) $N_2O_3 + R{\cdot}NH_2 \rightarrow R{\cdot}NH_2NO^+ + NO_2^-$

(8) Nitrosyl cpd. $+ R{\cdot}NH_2 \rightarrow R{\cdot}NH_2{\cdot}NO^+ +$ Nitrosyl-ion acceptor $(Cl^-, Br^-, HClO_4, HNO_3)$

(9) $R{\cdot}NH_2{\cdot}NO^+ \rightarrow R{\cdot}N_2^+ + H_2O$

Reaction (9) consists of the following steps—

$R{\cdot}NH_2{\cdot}NO^+ \rightarrow R{\cdot}NH{\cdot}NO + H^+$

$R{\cdot}NH{\cdot}NO \rightarrow R{\cdot}N_2OH$

$R{\cdot}NH_2OH \rightarrow R{\cdot}N_2^{+\cdot} + {\cdot}OH^-$

$H^+ + OH^- \rightleftharpoons H_2O$

Dependence of Bromo-Ion Catalysis of Diazotisation
H. Schmid and M. G. Fouad
Ibid., 631–638 (Sept. 1957)

Supplementing the rate law proposed by Schmid and Schubert (cf. *Z. Elektro-chem.*, **43**, 628 (1933))—

$$\frac{d\,(C_6H_5{\cdot}N_2^+)}{dt} = k\,[C_6H_5{\cdot}NH_2^+]\,[HNO_2]\,[Br^-]$$

the velocity coefficients of the Br^- catalysis of the diazotisation of aniline at 0°, 15°, and 25°c. are now reported. The time-determining reaction is the transfer of the nitrosyl ion from NOBr to the amino nitrogen of the unionised aniline. The data indicate that NOBr is more readily polarisable than NOCl. H.H.H.

Mechanism of Azo Coupling
A. A. Kharkharov
Trudy Leningrad. Tekstil. Inst., (5), 188–193 (1954): *Referat. Zhur., Khim.*, Abstr. No. 28889 (1956): *Chem. Abs.*, **52**, 9979 (25 June 1958)

Azo coupling requires the presence in the azo component of a reaction centre having high electron density. The azo component can be a substance having the CH_2 group with active H atoms. The activity of CH_2, CH_3 and CH groups with relation to azo coupling, increases with increase in positive charge of C atoms bound directly to these groups. The amount of this change is determined by the strength and number of electron-acceptor and electron donor atoms or groups linked with these C atoms directly or by conjugated bonds. Presence in the diazo component of an electrophylic substituent accelerates the reaction and a nucleophilic substituent retards it. C.O.C.

^{14}C-Labelled 4-Dimethylaminoazobenzene—II
O. Hromatka, L. Petzelbauer, and E. Broda
Mh. Chem., **88**, 317–319 (June 1957)

The preparation of 4-^{14}C-4-dimethylaminoazobenzene from diazotised aniline and 1-^{14}C-dimethylaniline is

described, and also the synthesis of a 4-dimethylamino-azobenzene in which all the carbon atoms of both benzene nuclei are labelled with ^{14}C. H.H.H.

Aromatic Diazo and Azo Compounds. XXVI— Hydrolysis and Ammonolysis of the Amino Group in Aminoazo Dyes; the Effect of Bisulphite

Z. J. Allan and J. Podstata.

Chem. Listy, **52**, 290–301 (1958): *Chem. Abs.*, **52**, 10986 (10 July 1958)

The rates of hydrolysis of the NH_2 group in azo dyes having α-naphthylamine (I) and its derivatives as end components were determined by colorimetric and chromatographic methods. E.g. at 20·6°C. on 5N HCl the dye aniline-2:5-disulphonic acid → 1-naphthylamine-7-sulphonic acid had a half-time of hydrolysis of 25·3 days, in 0·1N HCl 4·15 days and in aq. Na_2CO_3–$NaHCO_3$ at pH 10·4 > 210 days.

All dyes of this type reacted readily with $NaHSO_3$ with replacement of NH_2 by OH. Thus the monoazo compound *p*-nitroaniline → I in 1 hr. at 40°C. in aq. pyridine with $NaHSO_3$ gave the dye *p*-nitroaniline → α-naphthol, which in boiling pyridine with $NaHSO_3$ and gaseous NH_3 reverted to the original dye *p*-nitroaniline → I.

The theoretical background is discussed. E.S.

Separation Component in the Synthesis of Organic Dyes

I. Reichel and A. Balint

VI— Behaviour of the 1:5-Naphthylenedisazo System

Studii şi Cercetări Ştiinţifice, Timişoara, Seria Ştiinţe Chimice, **4**, 33–49 (July–Dec. 1957)

Owing to a break in the conjugate systems of dyes in the 1:4- and 1:5-naphthylenedisazo series, their colours correspond to intramolecular mixtures of those of the separated systems on each side of the break; i.e. each complete conjugate system acts as an independent component with respect to colour.

VII— Absorption Spectra of Some Azo Dyes containing the Central 1:4- and 1:5-Naphthylenedisazo systems

Ibid, 51–63

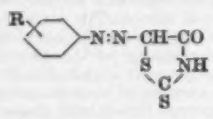
Spectral absorption curves are given for symmetrical and asymmetrical dyes of the above types and also of dyes representing the separate yellow and blue structural components of the asymmetric dyes. To obtain comparable spectra, dyes were chosen with salicylic acid as yellow component, and 1:8-aminoazonaphthol-2-*p*-nitrophenylazo-3:8-disulphonic acid as blue component. The neutral aq. soln. of the sodium salts of the dyes are blue, and this is displaced by alkali towards green, whereas acidification displaces the colour towards red–violet as far as violet; greens are obtained on cotton and wool. The resultant colours of these disazo dyes are due to the intramol. mixture of the colour effects of the individual sections of the mol. as divided by the break in the continuity of the conjugate system. H.H.H.

Synthesis and Transformations of Thiazolidine Derivatives. I— Preparation of Azorhodanines

A. P. Grishchuk and S. N. Baranov

J. Gen. Chem. U.S.S.R., **28**, 896–901 (April 1958)

The reaction between diazonium salts and rhodanine has been investigated and the preparation of 8 hitherto unknown azorhodanines of general formula—



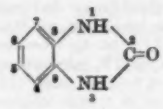
(R = H, O·CH_3, O·C_2H_5, $N(CH_3)_2$, $N(C_2H_5)_2$, or COOH) is described. G.J.K.

Derivatives of Glyoxalines. XX— Amino Derivatives of Benziminazolone

L. S. Efros and A. V. El'tsov

J. Gen. Chem. U.S.S.R., **28**, 941–945 (April 1958)

Compounds of the type—



(I)

have been treated with aromatic diazonium compounds. Whereas 5-amino-6-methylbenziminazolone does not couple, 5-amino-4:7-dimethylbenziminazolone reacts to give azo dyes. These results confirm the authors' previous conclusion on bond fixation in the molecule: the double-bond character of the 5–6 linkage is increased, whereas that of both the 4–5 and 6–7 linkages is decreased. G.J.K.

Monoazo Dye Derivatives of 1:2-Dimethylbenziminazole

M. T. Le Bris and H. Wahl.

Compt. rend. Acad. Sci. Paris, **245**, 2058–2060 (1957): *Chem. Abs.* **52**, 9086 (10 June 1958)

Coupling diazo compounds with 2-methylbenziminazolium salts gives disazo dyes in which 2 azo groups are attached to the same C atom. Monoazo compounds are prepared by indirect methods. Thus 1:2:3-trimethylbenziminazolium salts with *p*-nitrosodimethylaniline and K_2CO_3 in C_2H_5OH gives the corresponding azomethin (I), which on hydrolysis with HCl and addition of phenylhydrazine gives phenylazo-2-methylene-1:3-dimethylbenziminazoline.

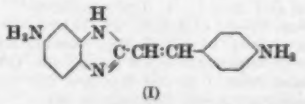
In another approach, 1:2-dimethylbenziminazoline, oxidised with SeO_2 in toluene at 95°C. gives 1-methyl-2-benziminazolecarboxaldehyde (II). Heating II in dil. HCl with phenyl-, *p*-nitrophenyl-, and 2:4-dinitrophenylhydrazine gives the hydrazones, which are easily *N*-methylated to yield alkylated hydrazones. Heating II with dimethyl sulphate or CH_3I gives I. E.S.

Diamines of the Benziminazole Series. II— Substantivity of Benziminazole Disazo Dyes

B. A. Porai-Koshits and Ch. Frankovskii

J. Gen. Chem. U.S.S.R., **28**, 934–938 (April 1958)

Diamines of the type—



(I)

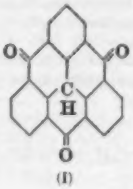
are tetrazotised and coupled with two molecules of the same α-naphthol, α-naphthylamine, or 1:8-aminonaphthol to give direct cotton dyes. Substantivity of these dyes is related to the presence or absence of conjugation between the terminal amino groups in I, and this relationship is not unduly affected by the character of the coupling components (e.g. J acid, H acid, or Chicago acid) used. G.J.K.

Substitution in the Condensed Triphenylmethane Triketone System

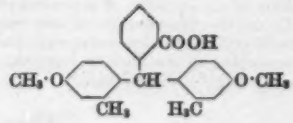
I. Reichel

Studii şi Cercetări Ştiinţifice, Timişoara, Seria 1 (*Ştiinţe matematice, fizice, chimice şi tehnice*), **2**, 91–96 (Jan.–Dec. 1955)

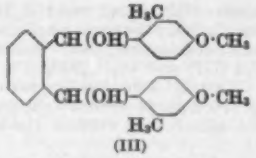
The possibility of introducing two methoxyl groups into condensed triphenylmethane–triketone (I) has been explored, and although the constitution of the final product has not yet been firmly established owing to crystallisation difficulties, the compounds 2:2′-dimethyl-4:4′-dimethoxytriphenylmethane-2″-carboxylic acid (II) and *o*-phenylenebis-(2-methyl-4-methoxyphenyl)- carbinol (III) have been prepared.



(I)



(II)



(III)

H.H.H.

Tautomerism of 9-Aminoacridine
A. K. Sukhomlinov
J. Gen. Chem. U.S.S.R., **28**, 1038–1045 (April 1958)

The absorption spectra of 9-aminoacridine and 10-methyl-9-iminoacridine in conc. H_2SO_4, in ethanolic HCl, in non-aq. and aq. dioxan, and in other solvents show that there is no tautomerism in 9-aminoacridine.

G.J.K.

Azo Derivatives of Cyanine Dyes
A. I. Kiprianov and I. P. Fedorova
J. Gen. Chem. U.S.S.R., **28**, 1023–1031 (April 1958)

From the quaternary salts of 2-methyl-5- and 2-methyl 6-arylazobenzothiazoles have been prepared and characterised a series of symmetrical and unsymmetrical thiacyanines and styrenes with an arylazo group in the benzene nucleus. The absorption spectra of these dyes in different solvents give only one intense band, showing that the azo substituents and polymethin chain form a common conjugated system. Compared with the arylazo grouping in the 6-position, the 5-substituted derivatives have an absorption band at shorter wavelengths.

G.J.K.

Polymethin Dyes. I—Comparison of Several Vinylogous Series in which Polymethin Chains are terminated by Aryl Groups
W. B. Tuemmler and B. S. Wildi
J. Amer. Chem. Soc., **80**, 3772–3777 (20 July 1958)

An account of the preparation and absorption spectra of four series of polymethin dyes having polymethin chains terminated by aryl groups. The x, y, and x' bands in the spectra were identified by application of principles known from triphenylmethane and cyanine dyes. When conjugation in the chain is extended there is a large bathochromic shift of the y-band as well as of the x-band.

C.O.C.

Effect of the Degree of Dispersion and Nature of the Adsorbent on the Spectral Curve of Absorption of Sensitising Dyes in the Adsorbed State.
Z. V. Zhidkova and Y. M. Suss.
Zhur. nauch. i priklad. fot. i kinematograf., **3**, 25–33 (1958):
Chem. Abs., **52**, 10774 (10 July 1958)

The intensity of light diffusely reflected from powdered Al_2O_3, $Al(OH)_3$, silica gel, diatomite, two types of glass, and quartz in presence of adsorbed layers of 1:1′-diethyl-2:2′-cyanine iodide (I) or 3:3′-dimethyl-9-ethyl-4:5:4′:5′-dibenzothiacarbocyanine chloride (II) was determined in the 400–700 mμ. range. Data are plotted for adsorbents of particle diameter equal to 1–2, 44–53, 105–125, 210–250 and 1000–1190 μ. In aqueous or alcoholic solution I has absorption maxima at 460, 490 and 524 mμ. in the adsorbed state the 460 mμ. maximum is not seen but a new maximum appears at 570–588 mμ. corresponding to the aggregate form of I. In aqueous solution II has absorption maxima at 510, 540 and 578 mμ. as well as a maximum at 655 mμ. corresponding to the aggregate form of II. The particle size of the adsorbent had no effect on the absorption spectra of I and II. 20 references.

C.O.C.

Synthesis of Phenylpyrazolone Derivatives
K. Itano

X—Absorption Spectrum of Phenylpyrazolone Azomethin Derivatives
Yakugaku Zasshi, **78**, 152–155 (1958)

Visible absorption spectra were measured of 30 phenylpyrazolone derivatives in methanol and examination made of the effect of a substituent R^2 in 1-position of the pyrazolone ring on the absorption, as well as that of R^1 in 3-position. When R^2 is an electron acceptor there is a tendency for increased contribution of the ionic resonance structure in the excited state and for the maximum absorption of the x-band to shift to a longer wavelength; when R^2 is an electron down, it shifts to a shorter wavelength.

XI—Notation of Colour of Phenylpyrazolone Azomethin Derivatives on the C.I.E. Colour Chart
Ibid., 155–158

The transmitted colour of the methanol solution was compared with the C.I.E. colour chart and observations made on the relationship between structure and dominant wavelength. The trichromatic coefficient was calculated by the wt. ordinate method, using C light source. When the substituent in 1-position of the pyrazolone ring is an electron acceptor, the dominant wavelength becomes longer, while it shortens when the substituent is an electron donor.

XII—Notation of the Colour of Magenta Dyes for Subtractive Colour Photography on the C.I.E. Colour Chart
Ibid., 159–163

Twenty-nine phenylpyrazolone dyes were condensed with p-$(C_2H_5)_2NC_6H_4NH_2$ to pyrazolone azomethin dyes on photographic film and trichromatic coefficients calculated from their spectrometric transmission rate. These were compared with the dominant wavelengths calculated from the ultraviolet and visible absorption spectra of the dyes in methanol. The dominant wave length of the dyes formed on a film is generally more on the shorter wavelength side than that in methanol, but the effect of each substituent in the dominant wavelength and the order of the shift of the dominent wavelength among these compounds approximated to those in methanol.

XIII—Solvent Effect on the Absorption Spectra of Phenylpyrazolone Azomethin Derivatives. 1.
Ibid., 164–167

The solvent effect on the ultraviolet and visible absorption spectra of 14 pyrazolone azomethin dyes was examined. They either possessed a substituent containing the SO_3NH group in the 3-position of the pyrazoline ring or had no such substituent. In both cases the maximum wavelength of the x-band tended to shift to a longer wavelength with increase of dielectric constant. Those with SO_3NH in the 3-position greatly affected the resonance system by dissociation of the SO_3NH group in a basic buffer solution and the band made a hypsochromic shift.

XIV—Solvent Effect on the Absorption Spectra of Pyrazolone Azomethin Derivatives. 2.
Ibid., 167–171

Effect of solvents of widely varying dielectric constants on the ultraviolet and visible absorption spectra of 12 pyrazolone azomethin dyes was examined. The maximum absorption of the x-band generally made a bathochromic shift with increasing dielectric constant, while that of compounds having a substituent containing SO_3NH in the 3-position of the pyrazolone ring made a hypochromic shift when the amount of water in the solvent exceeded a certain limit. Therefore a transition curve with molecular extraction at 550 mμ. clearly agreed with the dissociation curve. Compounds assumed to have steric hindrance had a larger dissociation constant and were less likely to be affected. Compounds having a substituent containing SO_3NH in the 1-position had smaller dissociation constants than those without such substituent; this state of dissociation was not seen in compounds other than those having a substituent containing SO_3NH in the 3-position.

XV—Solvent Effect on the Absorption Spectra of Pyrazolone Azomethin Derivatives. 3.
Ibid., 172–176
Chem. Abs., **52**, 11010 (10 July 1958)

Effect of solvent pH on the ultraviolet and visible spectra of 19 pyrazolone azomethin dyes was investigated. The maximum absorption of the x-band in these compounds generally shifted to shorter wavelengths with increasing pH and comparison of extinction coefficients at 550 mμ. showed that these compounds dissociated between pH 8 and 14. Compounds having a substituent containing SO_3NH in the 3-position of the pyrazolone ring underwent a two step dissociation at pH 2·1–4·0 and pH 4–7. Comparison of the effect of these substituents by measurement of dissociation constants showed the order of dissociation at pH 8–14 is $CH_3OC_6H_4SO_3NH$-p > CH_3 > $C_6H_5SO_3NH$ > B_2NH, while for compounds assumed to have steric hindrance it was $CH_3OC_6H_4SO_3NH$-p > $H_2NC_6H_4SO_3NH$-p > $CH_3C_6H_4SO_3NH$-p. Compounds having a substituent containing NH_2SO_3 at the 1-position of the pyrazolone ring had smaller dissociation constants than

those having no such substituent. Compounds assumed to have steric hindrance had larger dissociation constants than those without such hindrance and are less likely to be charged by the solvent pH. C.O.C.

Modified Primuline (C.I. 49000) Fusion and Separation of the Products by Alcoholic Extraction
J. Reichel, R. Palea, S. Iacobescu, and G. Gruescu
Studii şi Cercetări Ştiinţifice, Timişoara, Chimie, 1, 47–56 (Jan.–Dec. 1954)

Improvement in the ratio of dehydro-*p*-toluidine to Primuline base from 1:2 to 1:1 is obtained when the Primuline fusion is carried out in molten naphthalene at 210°C. After removal of the naphthalene by superheated steam at 250°C., dehydro-*p*-toluidine is extracted from the residual product with alcohol at 45–50°C. H.H.H.

Colour and Constitution. V—Indaniline Dyes
S. Hünig and P. Richters
Annalen., **612**, 282–288 (1957): *Chem. Abs.*, **52**, 11054 (10 July 1958)

With increase in λ of the absorption maximum of indaniline dyes from *o*-cresol and *NN*-aralkyl-*p*-phenylenediamine the polarographic half-wave potential of the diamine increases. C.O.C.

Substituent Effects. IV—Coupling of *NN*-Dialkylquinonediimonium Salts to yield Indaniline Dyes
S. Hünig and P. Richters
Annalen, **612**, 272–282 (1957): *Chem. Abs.*, **52**, 11044 (10 July 1958)

Spectrophotometric measurements of the concentration of dye were used to determine the rate of coupling of eight *NN*-dialkylquinonediimonium diperchlorates with 4-chloro-2-methylphenol at pH 6·3. Substituents which increased the polarographic half-wave potential of the *p*-phenylenediamines (from which the quinonediimonium compounds were formed) also increased the rate of coupling. Thus the morpholino derivative gave the fastest reaction followed by *NN*-dimethyl > *NN*-diethyl > piperidino > *N*-pyrrolidyl. Substituents in the 2-position of the 4-(*NN*-dimethyl) quinonediimonium diperchlorate increased the rate of coupling in the orders CH_3, H, Br, Cl. C.O.C.

Formation of Azine Dyes in Colour Development. II—Structure of the Azine Dyes from the Arylsulphonyl Derivatives of *m*-Diamines of the Benzene Series
I. V. Aleksandrov
Zhur. nauch. i priklad. fot. i kinematog., **2**, 432–436 (1957): *Chem. Abs.*, **52**, 9823 (25 June 1958)

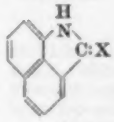
An account of work which proves that in forming azine dyes from arylsulphonyl derivatives of amino compounds, closing of the azine ring is accompanied by detachment of the arylsulphonyl residue. A sequence of reactions is given for the formation of azine dyes from *p*-phenylenediamine and arylsulphonyl derivatives of 2:4-tolylenediamine in colour development. The higher rate of formation of the dye 3-diethylamino-7-aminophenazine from 3-(4-tolylsulphonamido)aniline and 4-aminodiethylaniline sulphate compared with that using *m*-phenylenediamine and 4-aminodiethylamidine sulphate is caused by the activating effect of the arylsulphonyl group. C.O.C.

Chemotherapeutic Dyes. III—Synthesis of Substituted Aminophenothiazines
A. B. Sen and R. C. Sharma
J. Indian Chem. Soc., **35**, 202–204 (March 1958)

2-Aminothiophenol has been condensed with 5-methyl- and 5-ethyl-, with 6-carbomethoxy-, 6-carboethoxy-, 6-phenyl- and 6-methyl-2:4-dinitrochlorobenzene, and with 4-carboethoxy- and 4-*p*-nitrophenyl-2:6-dinitrochlorobenzene to give diphenyl sulphide compounds. With the exception of the first and the last, these, on treating with dilute alkali, were converted to the nitrophenothiazines and catalytically reduced to the corresponding amino compounds. C.H.R.

Thionaphthostyril Dyes
N. S. Dokunikhin and L. A. Gaeva
Khim. nauka i prom., **3**, 126–127 (1958): *Chem. Abs.*, **52**, 11427 (10 July 1958)

Thionaphthostyril (I) (1 g.) and 6-chloro-2:3-dihydro-3-oxothianaphthene (1 g.) were heated until no more H_2S came off and were then treated with boiling ethanol, 2% NaOH and alkaline $Na_2S_2O_3$ to yield $C_{19}H_{10}ONClS$ (II) (0·95 g.). I with 6-ethoxy-3-oxothianaphthene or 2:3-dihydro-4:5-benzo-3-oxothianaphthene, similarly treated, gave $C_{21}H_{10}NO_2S$ (III) and $C_{23}H_{13}NO_3$ (IV) respectively. I (1 g.) and 1-*p*-tolyl-3-methyl-5-pyrazolone (0·1 g). heated slowly to 180°C., kept at that temperature for 1 hr. and then treated with boiling ethanol yielded $C_{22}H_{14}N_2O$ (V).



(V) X = :C·C(CH$_2$):N·N(C$_6$H$_4$CH$_3$–4)·CO

(VI) X = :C(CN)COC$_6$H$_5$

(VII) X = 4-oxo-1(4*H*)naphthylidene

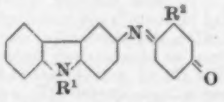
I (1 g.) heated 11 hr. at 140–145°C. with organoacetophenone (0·8 g.) and trichlorobenzene (4 ml.), cooled, and filtered, yielded after recrystallisation from ethanol $C_{29}H_{12}N_2O_2$(0·55 g.) (VI). Heating I (1 g.) with 1-naphthol (0·77 g.) for 14 hr. at 180°C. and extracting with hot ethanol (400 ml.) yielded $C_{21}H_{13}NO$(0·7 g.). These compounds after sulphonation with 5% oleum dyed wool and silk: II red-violet; III bright red; V orange-brown; VII blue. The absorption spectra had the following bands II 524, 559; III 395, 512·5, 546; IV 355, 422, 447, 527, 566; V 350, 368, 471; VI 290, 366, 459, 477; VII 533, 575, 622 mμ. The products could be obtained directly from naphthostyril by heating it in a suitable solvent with P_2S_5 before adding the other component. C.O.C.

Sulphuration of Organic Compounds in a Rotary Kiln
M. Russocki
Przemysł Chem., **37**, 46–47 (1958): *Chem. Abs.*, **52**, 12401 (25 July 1958)

Anthracene (18·4%), S (73·3) and anhydrous Na_2SO_4 (8·3) were ground in a ball mill and melted in an experimental rotary kiln. The kiln, 100 cm. in diameter and 50 cm. long, made of stainless steel, revolved at 10 r.p.m. and was heated with three burners. The exit gases, containing H_2S and CO_2 at 250°C., passed a cyclone before being liberated to the atmosphere. The products were olive dyes the exact hue depending upon the conditions of melting. C.O.C.

Sulphur Dyes from Carbazole–Indophenols
S. Moriwaki, O. Manabe, and H. Hiyama
Kôgyô Kagaku Zasshi, **59**, 1092–1093 (1956): *Chem. Abs.*, **52**, 10583 (25 June 1958)

Three indophenols of formula—



(R^1 = C_2H_5, H, and H; R^2 = H, H, and CH_3) were treated with S and Na_2S in butanol to yield dark blue, blue and reddish blue sulphur dyes respectively. The compound—

was similarly treated but could not be completely sulphurised, the product only yielding pale greenish blues on cotton. C.O.C.

Fluorescent Organic Materials and Chemical Constitution
L. Herforth
Wiss. Ann., **5**, 744–753 (1956): *Chem. Abs.*, **52**, 10861 (10 July 1958)

Review, 27 references. C.O.C.

Fluorescent Brightening Agents
R. S. Higginbotham and F. W. Thomas
Nature, **181**, 1437–1439 (24 May 1958)

A brief account of fluorescent brightening agents covering the properties required for use on textiles, their mode of action, chemical constitution of typical agents, measurement of efficiency, and the effects of exposure and storage on treated textiles. W.K.R.

Quenching of Fluorescence. III—Quenching of the Fluorescence of Fluorescent Brightening Agents
K. Weber and Z. Skuric
Croat. Chem. Acta., **29**, 115–125 (1957): *Chem. Abs.*, **52**, 10728 (10 July 1958)

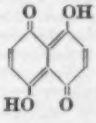
The intensity of fluorescence of Blankophor R and B, Ultraphor WT, Uvitex G3, RBS and RS, Leucophor WS and B (C.I. Fluorescent Brightening Agent 30, 32, 48, 42, —, 41, 52 and 50) deposited on Whatman No. 1 paper from organic solution was investigated before and after drying. Anions of inorganic salts (I^-, CNS^-, Br^-, NO_3^-) enhanced fluorescence while cations of heavy metals (Cu^{++}, Co^{++}) had an adverse effect as had hydroquinone and pyrogallol. Phenol had a quenching effect in solution only. Aniline sulphate had no effect. Acid and basic dyes had a quenching effect in very small concentrations. The effect of the quenching substances is interpreted by use of the Stein–Volmer equations (*Chem. Abs.*, **14**, 2885 (1920)). C.O.C.

Enhancement of Phosphorescence Ability upon Aggregation of Dye Molecules
E. G. McRae and M. Kasha
J. Chem. Phys., **28**, 721–722 (1958): *Chem. Abs.*, **52**, 11567 (25 July 1958)

Aggregation (dimers and polymers) of dye molecules may lead to important change in the emission spectra, in that the quantum yield of phosphorescence may be enhanced at the expense of fluorescence. Under conditions favouring phosphorescence at the expense of fluorescence it is most probable that it is the triplet levels, rather than the singlet levels, which are directly involved in ultimate energy transfer from the dye to the substrate. C.O.C.

Crystal Structure of Naphthazarin
G. A. Gol'der and G. S. Zhdanov
Doklady Akad. Nauk S.S.S.R., **118**, 1131–1133 (21 Feb. 1958)

From the electron-density map the bond-lengths and bond-angles in the molecule of naphthazarin are calculated. These results agree with a centrosymmetric structure.



G.J.K.

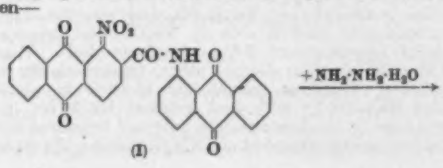
Absorption Spectra of Aminoanthraquinones
H. I. Stonehill
J.S.D.C., **74**, 585 (Aug. 1958)

Coplanarity and Conjugation of 1:4-Dianilinoanthraquinone and 1:4-Dimesitylaminoanthraquinone
N. S. Dokunikhin, G. A. Gol'der, and G. S. Zhdanov
Doklady Akad. Nauk S.S.S.R., **119**, 87–89 (1 March 1958)

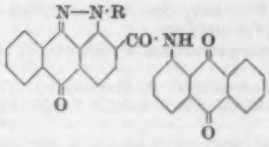
Unit cell dimensions and space group of 1:4-dianilinoanthraquinone (I) and 1:4-dimesitylaminoanthraquinone (II) have been determined. On the basis of packing considerations only, the authors suggest that I exists with the benzene nuclei almost coplanar with the anthraquinone system and that steric hindrance is absent; on the other hand, in II the benzene nuclei are rotated through approx. 90° from the plane of the anthraquinone system, and steric inhibition of conjugation between the benzene nucleus and the anthraquinone nucleus occurs. G.J.K.

Interaction of Hydrazine with Nitroanthraquinones. II—Synthesis of Vat Dyes derived from Pyrazoleanthronecarboxylic Acid
N. S. Dokunikhin and I. D. Pletneva
J. Gen. Chem. U.S.S.R., **28**, 1019–1022 (April 1958)

The preparation of II from I and hydrazine hydrate is given—

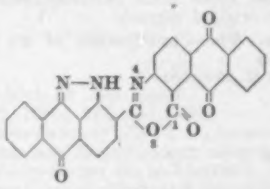


(I)



(II)

(R = H, CH_3), Hydrolysis of the amide linkage in II and formation of the corresponding acid chloride (III) are discussed. III reacts with 1-aminoanthraquinone-2-carboxylic acid, in the presence of $AlCl_3$, to give IV—

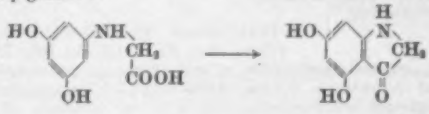


(IV)

the latter reacting with bases, e.g. ammonia or methylamine, and replacing the oxygen atom in the 2-position by an N·R^1 group (R^1 = H, CH_3). G.J.K.

Non-enzymatic Browning Reaction
H. Rinderknecht and L. Jurd
Nature, **181**, 1268–1269 (3 May 1958)

Solutions of phloroglucinol and glycine buffered at pH 6, 7, and 8 rapidly darken on storage, but solutions of phloroglucinol alone, under the same conditions, darken only slightly. It is suggested that the phloroglucinol condenses with the glycine to give precursors of a melanin-type pigment—



E.V.T.

Action and Emission Spectra of the Luminescence of Green Plant Materials
G. Tollin, E. Fujimori, and M. Calvin
Nature, **181**, 1266–1267 (3 May 1958)

Study of the spectral characteristics of the delayed light emission of green plant materials shows that the emission from both *Chlorella* and spinach chloroplasts results from the transition between the first excited singlet state and the ground state of chlorophyll, but for *Nostoc* the light emitted originates in phycocyanin. In the first two cases energy can be efficiently transferred from carotenoids to chlorophyll, but in *Nostoc* the energy transfer is much lower. E.V.T.

Inadequacy of the Concept of two Forms (Fluorescent and Non-fluorescent) of Chlorophyll from the Spectral Properties of the Living Leaf
V. Gachkovskii
Doklady Akad. Nauk S.S.S.R., **119**, 333–335 (11 March 1958)

Both fluorescent and visible absorption spectra of chlorophyll in the living leaf and in solution (in ether or methanol) are given, and on the basis of these results the author concludes that the previous suggestion of two forms of chlorophyll is invalid. G.J.K.

Mechanism of Sensitisation of Redox Reactions using Chlorophyll (C.I. Natural Green 3) and its Analogues in the Presence of Deuterium Oxide
V. B. Evstigneev and V. A. Gavrilova
Doklady Akad. Nauk S.S.S.R., **119**, 125–128 (1 March 1958)

The effect is investigated of replacing the labile H in the reducer (ascorbic acid or phenylhydrazine) by D upon the rate of photochemical reduction of Methyl Red (C.I. Acid Red 2) in pyridine using chlorophyll as sensitiser. The presence of D inhibits the formation of the second reduced form. The authors conclude that sensitisation occurs by means of the formation of the primary electronic reduced

form of the sensitiser, the latter reacting immediately with the acceptor–oxidiser. G.J.K.

Primary Photoreduction Products of Chlorophyll Derivatives
V. B. Evstigneev and V. A. Gavrilova
Doklady Akad. Nauk S.S.S.R., **118**, 1146–1149 (21 Feb. 1958)

The spectral data of chlorophyll derivatives (protophaeophytin, bacteriophaeophytin, and haematoporphyrin) after irradiation with a visible light source (300-watt lamp) are given. The authors claim that photoreduction occurs in two stages, with the intermediate formation of a meta-stable reactive product of a semiquinone radical type. This unstable intermediate can revert to the original pigment. G.J.K.

Orcein Dyes. VI— Constitution of α-, β-, and γ-Amino-orcein
H. Musso and H. Beecken
Chem. Ber., **90**, 2190–2196 (1957): *Chem. Abs.*, **52**, 10091 (25 June 1958)

The structures of α-, β-, and γ-amino-orcein (obtained by chromatographic separation of the constituents of orcein (see C.I. Natural Red 28)) are shown to be racemic 7-amino-4:5-dimethyl-6-(4:6-dihydroxy-2-methylphenyl)-2-phenoxazone, and racemic *cis*- and *trans*-7-amino-4:5-dimethyl - 3:6 - bis(4:5 dihydroxy - 2 - methylphenyl) -2-phenoxazone, respectively. C.O.C.

Constitution of Polycladin, the Yellow Pigment of *Lepidophyllum Quadrangulare*
G. B. Marini-Bettòlo, S. Chiavarelli, and C. G. Casinovi
Gazz. chim. ital. **87**, 1185–1199 (1957): *Chem. Abs.*, **52**, 10066 (25 June 1958)

Evidence which establishes the constitution of polycladin as 4′:5-dihydroxy-3:3′:6:7-tetramethoxyflavone. C.O.C.

Fungus Pigments. IV— Aurantiacin, the Pigment of *Hydnum aurantiacum*
J. Gripenberg
Acta Chem. Scand., **10**, 1111–1115 (1956): *Chem. Abs.*, **52**, 10935 (10 July 1958)

H. aurantiacum yields a red pigment, provisionally called *aurantiacin*, 2:5-bis(benzoyloxy)-3:6-bis(*p*-hydroxyphenyl)-1:4-benzoquinone, which is the benzoate of the fungus pigment atromentin—

$$\overline{OC{\cdot}C(OH){:}C(p\text{-}HOC_6H_4){\cdot}CO{\cdot}C(OH){:}C}{\cdot}p\text{-}HOC_6H_4.$$

C.O.C.

Composition of Earths and Ochres from Western Bohemia. I— Artist's Earths
E. Slánský
Rozpravy Československ. akad. věd., Řada mat. a přírod věd., **68**, (i), 61–78 (1958): *Chem. Abs.*, **52**, 9879 (25 June 1958)

Examination of some earths used as pigments showed presence of quartz, muscovite and kaolin while one sample also contained gibbsite. 21 references. C.O.C.

Egyptian Blue (C.I. 77437)
G. Schippa and G. Torraca
Rass. chim., **9**, (6), 3–9 (1957): *Chem. Abs.*, **52**, 10604 (25 June 1958)

The ancient pigment known as Egyptian Blue, Pompeian Blue or Alexandria frit contain SiO_2, CaO and CuO. A pigment similar in appearance was prepared by heating silica gel, CaO and CuO at 840°C. A prolonged time of reaction favours production of crystals. Slight molar excess of CuO over CaO and slight excess of both oxides over SiO_2 gives the best product. Use of SiO_2 gel instead of quartz speeds up the reaction. C.O.C.

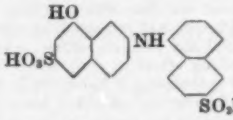
Colour Problem of Ruby
L. Singh
Nature, **181**, 1264–1265 (3 May 1958)

Orgel's hypothesis, that the colour of ruby (solid solutions containing < 8 mol. % Cr_2O_3 in Al_2O_3) arises from compression of the chromic ions, is criticised. The absorption band of the green chromic oxide shifts from 588 to 526 mμ. in red rubies, but the shift is gradual. The colour of the crystals is green at concn. down to 30 mol. % below this concn. it becomes greyish green, grey, greyish red, and then bright red, which remains stable below 8% molar. Changes in the lattice parameter and in the density of the solid support the concept of compression, but the alternative explanation suggested is that the uncoupled 3*d* electrons of the chromic ion are squeezed into "satellite" orbitals in the space surrounding the ions. E.V.T.

PATENTS

Preparation of *N*-(6-Sulpho-1-naphthyl)-γ Acid
Ciba *BP* 797,353

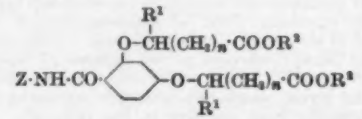
The reaction between γ acid or 2:8-dihydroxynaphthalene-6-sulphonic acid and 1-naphthylamine-6-sulphonic acid in presence of a sulphite gives the title product—



although reactions between isomers of these starting materials do not proceed smoothly. E.S.

Derivatives of 2:4-Dicarboxyalkoxy Benzamides as Colour Developers
Kodak *BP* 797,995

Compounds of formula—



(R^1 = H or Alk; R^2 = H, CH_3 a alkali metal; n = 0 or 1; Z = aromatic nucleus), e.g. 1-(2:4:6-trichlorophenyl)-3-[3-(2:4-dicarbomethoxymethoxybenzamido)benzamido]-5-pyrazolone, are useful photographic colour developers. C.O.C.

Diazotisation of 1:2:4 Acid
American Cyanamid Co. *USP* 2,812,321

By diazotising 1:2:4 acid (1-amino-2-naphthol-4-sulphonic acid) in presence of a surface active polyethylene oxide fatty acid ester, excessive thickening of the reaction mixture is avoided, thereby facilitating cooling and pumping, and making isolation of the diazo compound unnecessary when coupling with e.g. 1-naphthol-8-sulphonic acid. The preferred surface active agent is a condensation product of tall oil with ethylene oxide, having ca. 16 ethylene oxide units in a polyethylene oxide chain, and 10–20% on the weight of the 1:2:4 acid is used, with $CuSO_4$ and/or $ZnCl_2$ to minimise oxidation. E.S.

Synthesis of β-Carotene and its Dehydro-derivatives
F. Hoffmann–La Roche & Co. *BP* 797,635

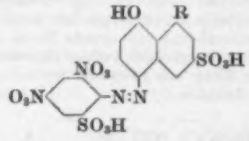
β-Carotene and other carotenoids can be obtained from vitamin–A aldehyde or vitamin–A_2 aldehyde in a simple synthesis of type $C_{20} + C_3 + C_{17} = C_{40}$ which involves preparation of C_{40}-(acetylenic diols) which are readily converted into carotenoids. Thus dry acetone-free acetylene is lead into a solution of Li in liquid NH_3 until reaction ceases. β-C_{17}-ketone in absolute diethylether is added and the mixture stirred with complete exclusion of moisture for 12 hr. NH_4Cl is then added and the ammonia evaporated off. Water is added to the remaining liquid, the ether layer separated, washed with water, dried with Na_2SO_4 and evaporated. The residual reddish oil is vigorously dried in a vacuum to yield crude β-C_{40}-(acetylenic carbinol) which can be purified by partition between petroleum ether and 90% aqueous methanol or by chromatography on aluminium oxide. This is dissolved in absolute alcohol, treated at 10–20°C. with a Grignard solution and heated under reflux for 1 hr. in an atmosphere of N, cooled with ice water and added at 20°C. to a solution of vitamin–A aldehyde in absolute diethyl ether and refluxed for 3–4 hr. in an atmosphere of N. The resulting solution is poured into 3 N-H_2SO_4 and ice, the ethereal layer separated, washed with aq. Na_2CO_3 and evaporated to yield asymmetrical β-C_{40}-(acetylenic diol). This is dissolved in absolute diethyl ether, treated slowly with a solution of lithium aluminium hydride in absolute diethyl ether at 10–20°C. and refluxed for 24 hr. in an atmosphere of N. The resulting mixture is poured into a 50/50 mixture of saturated aq. NH_4Cl and ice, the ethereal

layer separated, dried with Na_2SO_4 and evaporated. This yields crude β-carotene which can be purified by partition between solvents or by chromatography. C.O.C.

Indicator Dyes

H. Wenker *USP* 2,806,023

Dyes of formula—

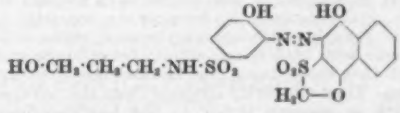


(R = sulphonic, sulphamide, *N*-(benzyl-substituted sulphamide and *N*-(lower alkyl)-substituted sulphamide) are particularly useful in the production of indicator papers. Thus filter paper impregnated with the K salt of 4-(2:4-dinitro-6-sulphonicphenyl azo)-1-naphthol-6:8-disulphonic acid shows significant change of colour giving almost instantaneous and clear differentiation between pH 4 and below (yellow), 5 (yellow green) 6, 7 (dark green), 8 and above (dark blue). The colours are pure and intense. C.O.C.

Violet and Blue Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, having an Oxathiol-S-Dioxide Ring System

FBy *BP* 796,759

Monoazo dyes made by coupling diazotised 2-aminophenol-4-sulphon-γ-hydroxypropylamide with 1:4-naphthohydroquinonemethylenesulphon ether (4′-hydroxy-naphtho (2′:1′:4:5) oxathiol-S-dioxide)—



are converted into their 1:2 metal-complexes; the Cr-complex gives reddish-blue on wool from a weakly acid bath, and the Co-complex a reddish-violet. The coupling component is made by the method of *BP* 731,495, comprising heating the addition product of 1:4-naphthaquinone and a suitable organic sulphonic acid. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Pyrazolone Dyes for Wool

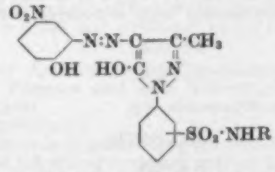
FBy *BP* 796,918

The complexes containing 1 atom of Cr or Co to 2 mol. of the monoazo compound 2-aminophenol-5-sulphonamide → 3-methyl-1-*m*-nitrophenyl-5-pyrazolone are respectively scarlet-red and yellow-brown dyes for wool, applied from neutral baths. E.S.

Metal(Cobalt)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

General Aniline *USP* 2,809,962

The metal complexes from 1 atom of Co and 2 mol. of monoazo compounds—



(R = H or lower Alk) dye wool, nylon etc. from neutral or weakly acid baths. Thus the monoazo compound 2-amino-4-nitrophenol → 3-methyl-1-*m*-sulphamylphenyl-5-pyrazolone is heated for 6 hr. at 90°c. with aq. $CoCl_2$. The product dyes wool beige of good light fastness. E.S.

Yellow Metal (Copper with Nickel)-complex Monoazo Pigments

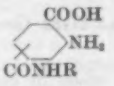
DuP *USP* 2,801,996

Yellow pigments are made by metallising monoazo compounds obtained by coupling a diazotised arylamine free of OH groups *ortho* to the NH_2 group, SO_3H and COOH groups, with a 4-hydroxy-2-pyridone compound, and metallising, first with an Ni salt and then with a Cu salt so that the resulting pigment contains 2 mole of monoazo compound to 1 atom of metal, the ratio of Ni to Cu being about 3 to 1. Thus, all parts being by weight, *p*-chloroaniline (128) is diazotised and coupled with 2:4-dihydroxyquinoline (169), and the suspension of monoazo compound so formed is heated to 80°c. and stirred with $NiSO_4 \cdot 6H_2O$ (151) in presence of aq. ammonia. After ca. 1½ hr. $CuSO_4 \cdot 5H_2O$ (84) dissolved in aq. ammonia is added and stirring continued at 80°c. for a further 1½ hr. The product is faster to bleeding into organic solvents than the similar pigment described in *USP* 2,396,327 prepared by metallising the same azo compound solely with an Ni salt. E.S.

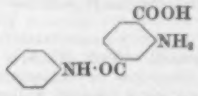
Monoazo Pigments

DuP *USP* 2,808,400

Amines of general formula—



(R = aryl of benzene or naphthalene series and may carry substituents such as Alk, OAlk, Hal, NO_2; CONHR is in the 4, 5 and 6 position relative to the COOH group) are diazotised and coupled with suitable coupling components, followed by laking with metal (especially Sr) salts to give pigments of good intensity, brightness and fastness to aq. alkalies. Thus 2-aminoterephthanilic acid—

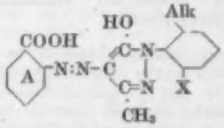


is dissolved in aq. NaOH containing $NaNO_2$, and run into dil. aq. HCl to give the diazo compound which is added to 3-hydroxy-2-naphthoic acid dissolved in aq. NaOH containing Na_2CO_3. The product is boiled with aq. $Sr(NO_3)_2$ in presence of Turkey Red Oil to give a red pigment. E.S.

Yellow Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, etc.

Ciba *BP* 797,046

The 1:2 Cr-complexes of monoazo compounds—



(the ring A may contain non-solubilising substituents; Alk is preferably C_2H_5; X = H, Alk, Hal, etc.) dye wool, nylon, etc. yellow from neutral or weakly acid baths. Thus the monoazo compound anthranilic acid → 1-*o*-ethylphenyl-3-methyl-5-pyrazolone is boiled for 24 hr. with aq. sodium chromosalicylate or with aq. CrF_3 in presence of sodium acetate. E.S.

Red and Brown Lakes of Monoazo Dyes Derived from Arylides of 3:2-Hydroxynaphthoic Acid.

Gy *BP* 797,850

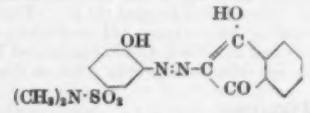
Anilinesulphonic acids containing a further SO_3H group or a COOH groups are diazotised and coupled with arylides of 3:2-hydroxynaphthoic acid, and the monoazo compound so formed precipitated as lakes with salts of Ca, Ba, Sr, Mn, Fe, Al, etc., to give pigments fast to bleeding into organic solvents. The diazo component preferably has SO_3H or COOH *ortho* to the NH_2 group. Thus aniline-2:5-disulphonic acid is diazotised and coupled with a soln. in aq. NaOH of 3:2-hydroxynaphtho-α-naphthylamide. After neutralising, a linseed oil emulsion is added followed by dropwise addition of aq. $CaCl_2$. The temperature is raised to 75–80°c. and the lake isolated after cooling overnight. It colours polyvinyl chloride red of good fastness to light, heat and migration. E.S.

Metallisable Monoazo Dyes for Wool, etc., from Indandione (1:3)

FBy *BP* 797,231

Sulphonamides or alkylsulphone derivatives of *o*-aminophenol are diazotised and coupled with indandione (1:3)

or its derivatives like indandione (1:3)-2-carboxylic acid which form indandione (1:3) under the conditions of coupling. The products may be metallised in substance, on the fibre or in the dyebath with metal (especially Co and Cr) salts. Thus 2-aminophenol-4-sulphondimethylamide is diazotised and coupled with a suspension of indandione (1:3) in aq. ammonia to give—

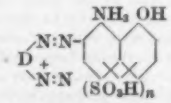


Heating with aq. ammoniacal $CoCl_2$ gives the metal complex which dyes wool red-brown from a neutral bath. E.S.

Black, Dark Blue and Dark Green Trisazo Dyes for Leather, etc.

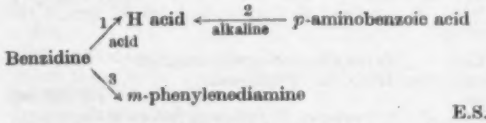
CFM *BP* 796,790

Trisazo compounds made by coupling a diazomonazo compound—



(D = diphenyl residue)

with a diazotised *m*- or *p*-aminobenzoic acid to give a diazodisazo compound, which is then coupled with a *m*-di-hydroxy- or -diamino- or -aminohydroxy-compound of the benzene series, gives intense, well-penetrated blacks on leather, and may also be used for blacks, dark blues, or dark greens on cellulose and animal fibres. An example is—



E.S.

Production of Stabilised Diazonium Salts of Improved Physical Form

General Aniline *USP* 2,809,189

The stable compounds obtained by combining diazonium salts with aryl or alkyl sulphonates, or with acid sulphate, or with $ZnCl_2$, sodium fluoroborate, etc. are produced in coarser particle size by carrying out the precipitation in presence of a suitable arylsulphonic acid, e.g. benzenedisulphonic acids, toluenesulphonic acids, xylenesulphonic acids or naphthalene di- and tri-sulphonic acids, giving improved filtering properties, drier press-cakes, and less dusty powders on drying. Thus to a filtered solution of diazotised 4-amino-2:5-dimethoxy-4′-nitroazobenzene is added a solution containing 0·5 mol. of $ZnCl_2$ and 20% on the weight of amine of xylenesulphonic acid. After salting out and standing 2 hr. the large reddish-brown crystals are filtered off, mixed with drying compounds, and dried. E.S.

Blue Sulphur Dyes

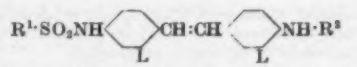
Allied Chemical & Dye Corpn. *USP* 2,801,995

Brighter blue dyeings on cotton or other cellulosic fibres are obtained from sulphurised 2:4-dinitro-4′-hydroxydiphenylamine dyes which have been treated under alkaline conditions with an alkali metal cyanide or alkaline-earth metal cyanide. Thus, the filter cake obtained by reacting a polysulphide, indophenol (5) and 2:4-dinitro-4′-hydroxydiphenylamine (95) is slurried with water (4000); 50% aq. NaOH (200), $Na_2S_2O_4$ (30) and NaCN (10) are added in that order and the mixture is air-blown at room temperature for 20 hr. Addition of NaCl (1000) precipitates the dye which gives bright blue dyeings directly from an Na_2S bath. If NaCN is omitted from this treatment products which dye very much duller blues are obtained. E.T.

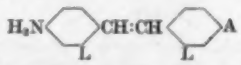
Fluorescent Brightening Agents

Ciba *BP* 797,427

Compounds of formula—



(L = sulphonic acid; R^1 = subst. or unsubst. Ar; R^2 subst. or unsubst. acyl, triazyl or carbamic acid residue containing no group imparting colour) e.g. 4-amino-4′-benzenesulphonamidostilbene-2:2′-disulphonic acid, have affinity for vegetable, animal and man-made fibres on which they show a violet blue to greenish yellow fluorescence in ultraviolet radiation. They are prepared by treating 1 mol. of a compound of formula—

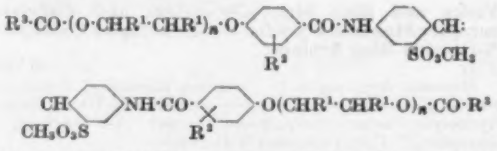


(A = NH_2 or NO_2) with 1 mol. of a compound of formula R^1SO_2 Hal, if necessary reducing it to NH_2 and then treating with a compound of formula R^2SO_2 Hal. C.O.C.

Fluorescent Brightening Agents

Fabrique de Produits Chimiques G. Zimmerli *USP* 2,806,054

Compounds of formula—

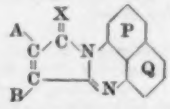


(R^1 = H or Alk; R^2 = Alk, oxyalkyl or alkoxy; R^3 = Alk, Ar, hydroaromatic or heterocyclic radical), e.g. a 4:4′-bis-(*p*-acyloxyethoxybenzoylamino)-stilbene-2:2′-disulphonic acid, have strong affinity for cellulose at 50°C. on which they show strong blue fluorescence in ultraviolet radiation. They are partly stripped from the cellulose by treatment in aqueous liquor in the neighbourhood of 100°C. C.O.C.

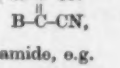
Coloured Polycyclic Compounds

ICI *BP* 799,352

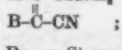
Polycyclic compounds for the mass coloration of plastics and synthetic fibres and of good fastness to light are of formula—



(X = Cl_2, Br_2, S or NH; A, B = subst. or unsubst. hydrocarbon radicals which may join to form *homo*- or *hetero*-cyclic rings which, in turn, may be substituted with Hal, Alk, alkoxy, aryl, aryloxy or NO_2 groups; nuclei P, Q may be substituted with Hal, alkoxy or an ethylene radical connected to the peri-C atoms). Methods of manufacture include (1) (where X = NH or S) heating 1:8-naphthylenediamine with a dinitrile, e.g.



or with a cyanothioamide, e.g.



(2) (where X = Cl_2, Br_2 or S) reacting the corresponding keto compound with PCl_5, PBr_5 or P_2S_5; (3) (where X = S) heating the dihydrogen compound with S. 17 examples are given. E.T.

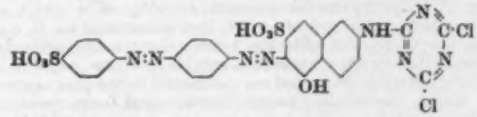
Reactive (Azo) Dyes having a Cyanuric Chloride Residue which Reacts with Cellulose

ICI *BP* 797,946

Azo dyes having at least one solubilizing group (e.g. SO_3H) and at least one amino group carrying as *N*-substituent a 1:3:5-triazine radical containing at least 1 Hal atom attached to a C atom of the triazine ring, react with cellulose in presence of an acid-binding agent, giving dyeings and prints fast to wet treatments. Such dyes may

be made; (a) by condensing a suitable aminoazo compound, e.g. 2-naphthylamine-4:8-disulphonic acid → *m*-toluidine, with 1 mol. of cyanuric chloride (I); (b) by condensing 1 mol. of a suitable amino-compound, e.g. J acid, with 1 mol. of I, and then coupling with a diazo compound; (c) by condensing 1 mol. of I with 1 mol. of a suitable diamine, e.g. *p*-phenylenediamine sulphonic acid, the product being then diazotised and coupled with a suitable coupling component.

Thus diazotised 4-aminoazobenzene-4′-sulphonic acid is diazotised and coupled with the condensation product of 1 mol. of I and 1 mol. of J acid, to give the disazo dye—



Cotton padded with an aq. soln. of this dye, dried at 80°C., immersed for 1 min. at 95°C. in 6% aq. NaOH saturated with salt, rinsed, immersed in cold 0·2% aq. acetic acid, rinsed, and scoured in hot soap and soda ash soln. is coloured red fast to washing. E.S.

Acid Milling of Vat Dyes
General Aniline *USP* 2,796,418

Coarsely crystalline or agglomerated amorphous vat dyes (1) are milled in a Werner–Pfleider type mill with Na_2SO_4 (0·5–5) and conc. H_2SO_4 (0·5–2) to give a doughy mass. The mass is then discharged into water (10) with agitation, the dye being recovered by filtration and washing acid-free. The dye is obtained in a suitable form for textile dyeing, printing, padding and mass coloration. It is claimed that this method has economic advantages and gives greater ease of handling by comparison with earlier methods, e.g. those of *USP* 2,334,812; 2,176,011; 2,402,167. E.T.

Carbon Black (C.I. Pigment Black 7)
United Kingdom Chemicals *BP* 799,145

Apparatus for preparing furnace black from gases, vapours or atomised liquids. C.O.C.

Electrolytic Treatment of Carbon Black (C.I. Pigment Black 6 and 7)
Godfrey L. Cabot *USP* 2,807,577

An aqueous slurry of carbon black is maintained in direct contact with the anode of an electrolytic cell but is completely isolated from the cathode by a membrane which is permeable to water and substances dissolved in it but is impermeable to the pigment. This increases the amount of O, S or other chemical complexes in the carbon black. The resulting change in surface properties can be very delicately controlled. C.O.C.

Iron Oxide Pigment
J. McNicholl *BP* 799,202

An alkali or alkaline earth carbonate is dry mixed with ferrous sulphate, both components of the mixture being of particle size < 200 mesh. The ratio of the two components varies from 1:4 to 4:1. The mixture is allowed to absorb atmospheric moisture for 4–7 days and is then tightly packed and stored for 3 weeks at room temperature. Finally it is dried and powdered to particle size < 200 mesh. C.O.C.

Basic Lead Carbonate for Producing Pearlescent Effects in Plastics and as a Substitute for Natural Pearl Essence
Argenta Products Co. *USP* 2,807,858

Carbon dioxide is passed through aqueous lead acetate (50% based on wt. of hydrated neutral lead acetate) at > 50°C. preferably at 85–100°C. and pH > 4·5 preferably about 5·5. This yields hexagonal crystals of particle size 5·0–30·0 μ. The crystals should be removed from the solution as soon as formed to prevent overcarbonisation causing lumping. The lustre of the product is enhanced when the crystals are dispersed in an organic liquid. They are heat resistant enough to enable them to be used to produce pearlescent effect in plastics and they can substitute natural pearl essence in many fields of use. C.O.C.

Basic Magnesium Chromate as Corrosion-Inhibiting Pigment
Associated Lead Manufacturers *BP* 798,969

Basic magnesium chromate having 5 mol. of MgO to 1 of CrO_3 added in small amounts to other pigments gives much better inhibition of corrosion and prevents blistering of the paint film when it is immersed in seawater. Thus light magnesium oxide is mixed with a main pigment, e.g. iron oxide, and then ground with an aqueous solution of CrO_3 and dried. C.O.C.

Silicon Dioxide (C.I. Pigment White 27)
Columbian Carbon Co. *BP* 797,374

A molten mass containing silicon, e.g. a melt of ferrosilicon, is treated with air or other oxygen containing gas which is preferably heated before coming into contact with the melt. The pigment is recovered by known methods from the effluent gases. C.O.C.

Silica (C.I. Pigment White 27)
Columbia-Southern Chemical Corpn. *USP* 2,805,956

A pigment of low water content is obtained by heating hydrated silica to effect controlled removal of water from it while controlling the conditions of heating so as to prevent excessive crystal formation. C.O.C.

Hydrophobic Silica (C.I. Pigment White 27)
General Electric Co. *USP* 2,805,958

Silica is treated with an alcohol in the presence of a strong acid, e.g. with HCl in butanol. C.O.C.

Titanium Dioxide (C.I. Pigment White 6)
DuP *BP* 799,205

The film fineness, gloss and colour characteristic of TiO_2 are improved by dry grinding it in presence of a little, e.g. 0·005–0·5% on the wt. of TiO_2, of triethanolamine and a little, e.g. 0·05–5·0% of an aerogel, e.g. silica aerogel, which eliminates metallic erosion during the grinding. C.O.C.

Titanium Dioxide Pigments (C.I. Pigment White 6) for Stoving Enamels
American Cyanamid Co. *USP* 2,804,374

The tendency of TiO_2 to cause enamels to darken and become dull on stoving can be markedly reduced if during the manufacture of the TiO_2 the titania hydrolysate is partly dried and then kneaded until a semi-pasty mass is obtained. This is then calcined and processed to the pigment as usual. C.O.C.

Ultramarine (C.I. Pigment Blue 29)
American Cyanamid Co. *USP* 2,806,802

Al silicate in suitable form, e.g. English China Clay, is added to aqueous sodium sulphide or polysulphide at ≮ 100°C. The clay dissolves immediately and a mixture of primary and secondary ultramarines precipitates. The primary ultramarine is converted into the secondary ultramarine of commerce by treatment with an oxidising agent. On filtering the slurry and drying a good quality ultramarine is obtained. C.O.C.

Weather-proof, Tinted, Zinc Sulphide Pigments
"Sachtleben" AG für Bergbau und Chemische Industrie *BP* 797,845

Compounds of Fe, Ni, Cu or Co are added to zinc sulphide pigments before calcining so that there is 0·1–2·0% (on wt. of Zn) of added metal. This prevents photolysis of the zinc sulphide to zinc sulphate and so obviates the disintegration of pigment films caused by the action of zinc sulphate. C.O.C.

Zinc and Cadmium Sulphides and Selenides
DuP *USP* 2,805,917

High yields of pure products are obtained by dissolving a Zn or Cd salt of a weak acid in an alkanol of 1–5 C, passing H_2S or H_2Se through the solution while keeping the liquor under reflux and recovering the metal chalcogenide. This is washed with additional alkanol and dried at 60–120°C. preferably under reduced pressure. C.O.C.

Sulphonation with Sulphur Trioxide–High-boiling Alkylated Benzene (III p. 704)
Zinc Stearate as Flatting Agent (V p. 716)
Polyethyleneglycols as Detergent Assistants (VII p. 717)
Present Trends in the Russian Manufacturing, Dyeing, and Printing Industries (VIII p. 718)
1:4-Dihydroxynaphthalene Monoalkyl Ethers in dyeing Vinylon (VIII p. 719)
Paper-coating Trends in the United States (XI p. 723)
Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XIV—3-Bromo- and 3:3′-Dibromo-benzidine as Redox Indicators (XIV p. 727)

Preparation and Analytical Properties of *NN*-Di(carboxymethyl)aminomethyl Derivatives of some Hydroxyanthraquinones (XIV p. 728)

Tannins. VII— Colorimetric Determination of D-Catechin in Catechin (C.I. Natural Brown 3) (XIV p. 728)

V—PAINTS; ENAMELS; INKS

Zirconia Enamels

Š. Vytasilová and V. Vytasil

Silikáty, **1**, 322–329 (1957): *Chem. Abs.*, **52**, 12352 (25 July 1958)

Review; 58 references. C.O.C.

Fluorescent Coloured Porcelain Enamel

B. R. Eichbaum

Ceram. Ind., **70**, (6), 92–93 (1958): *Chem. Abs.*, **52**, 12352 (25 July 1958)

Fluorescent coloured porcelain enamels have been produced by combining fluorescent pigments with two clear ground coat molybdic enamels which are fired at 730–830°C. Phosphorescent and electroluminescent enamels were obtained by adding a Cu-activated ZnS pigment and using a triple layer process. The matured enamels show coloured fluorescence under ultraviolet radiation. Other pigments used include UO_2, MoO_3, ZnS, wollastonite, willemite and proprietary mixtures. C.O.C.

PATENTS

Silica Gel Flatting Agent

W. R. Grace & Co. *BP* 798,621

Silica hydrogel is washed, dried and ground, being impregnated, at any stage after washing, with an inert, colourless, high m.p., microcrystalline wax which is insoluble in the solvent in which the flatting agent is to be used. This yields a flatting agent of improved flatting qualities which remains in suspension in varnish, etc. during long periods of storage. C.O.C.

Zinc Stearate as Flatting Agent

American Cyanamid Co. *USP* 2,809,121

Zinc stearate of good flatting power, i.e. of large average crystal size, is obtained if a little phthalic or maleic acid is present when the zinc stearate is formed. C.O.C.

Dual-purpose Tinting Base

Sherwin-Williams Co. *USP* 2,809,122

Tinting bases for both water-insoluble solvent-thinned enamels and water-thinned emulsion paints comprise a water-insoluble pigment, a volatile organic solvent at least a portion of which is both miscible with water and soluble in oleoresinous varnishes, and a liquid surface-active composition of which at least one component is non-ionic. The most suitable pigments are lead chromate yellows (C.I. Pigment Yellow 43) molybdate orange (C.I. Pigment Red 104); phthalocyanine blue (C.I. Pigment Blue 15), lamp black (C.I. Pigment Black 6), red iron oxide (C.I. Pigment Red 101), low absorption value ferrite yellow (C.I. Pigment Yellow 42) iron brown (C.I. Pigment Brown 6 and 7), Permansa Green (β-naphthol-iron complex), TiO_2 (C.I. Pigment White 6) and China clay (C.I. Pigment White 19). C.O.C.

Heat-resistant Coating Compositions containing Leafing Pigments

Allied Chemical & Dye Corpn. *USP* 2,803,610

Organosilanols of mol. wt. < 3000 are compatible with resinous rosin esters of polyhydric alcohols. Such a solution can be used as the vehicle for high temperature-, chemical- and weather-resistant finishes of the inorganic or metal leaf or powder pigmented types. The vehicle improves the leafing action of the pigments and yields a paint resistant to temperatures of 1000–1500°F. C.O.C.

Preserving Proteinaceous Coating Compositions, etc. from Microbiological Attack

BASF *BP* 797,526

A water-soluble polyamide-forming substance, or a polyvalent urethane (which may have an organic substituent on the N atom), or a carboxylic acid amide having an organic substituent on the N atom, or urea substituted on at least one N atom by an organic radical is a good antiseptic for use in proteinaceous coating compositions. It must contain no *N*-methylol group and should be $\geq 80\%$ on the dry weight of the protein. Suitable compounds are γ-butyro-lactam, amino lactones, amino carboxylic acids, etc. C.O.C.

Unsymmectrical Dimethylhydrazine for Preventing Paints, etc. Skinning during Storage

Olin Mathieson Chemical Corpn. *BP* 799,922

Addition of 0·05–0·5% by wt. of unsymmetrical dimethylhydrazine, $(CH_3)_2NNH_2$ does not interfere with the drying properties of a paint but does prevent skin formation while the paint is in store. C.O.C.

Iodine Complexes of Copolymers of *N*-Vinyl Pyrrolidones and Vinyl and Vinylidene Compounds containing No Nitrogen

General Aniline *BP* 799,622

Copolymers of 60–90% by wt. of a *N*-vinyl pyrrolidone, e.g. *N*-vinyl-2-pyrrolidone, and 40–10% of a vinyl or vinylidene compound of < 8 C and containing no N, e.g. vinyl or vinylidene chloride, readily form iodine adducts. These adducts are very stable, soluble in water, capable of forming flexible films and very effective in the preparation of marine, antifouling paints, termite and fungi resistant coatings, etc. C.O.C.

VI—FIBRES; YARNS; FABRICS

Books about Textiles— A Classified List of Recently Published Literature

Dyer, **120**, 95–97 (18 July 1958)

Desulphurisation of Viscose Rayon Fibre

G. G. Finger, A. B. Pakshver, and E. M. Mogilevskii

Tekstil. prom., **18**, 17–19 (May 1958)

The rate of desulphurisation of viscose rayon fibre by non-aq. solvents, by water, and by acids depends on the rate of diffusion (*a*) of the reagent into the fibre and (*b*) of sulphur out of the fibre. The diffusion coefficient of desulphurisation increases sharply with increasing swelling of the fibre and is most pronounced in alkaline solution. The rate of diffusion is smaller in oriented (stretched) than in non-oriented fibres owing to the more densely packed molecular structure. The presence of surface-active agents greatly promotes desulphurisation in weakly acid and neutral solutions, which is attributed to the dispersing action on sulphur. G.J.K.

Defective Wool produced by Sheep-dips

C. Earland

J.S.D.C., **74**, 545 (July 1958)

Application of "Chromatoelectrophoresis" to the Analysis of various Morphological Fractions of Wool.

J. Derminot

Bull. Inst. Text. France, (72), 69–73 (Feb. 1958)

Paper chromatography in butanol or phenol, followed by electrophoresis in a second dimension using veronal buffers, gives good resolution of basic and acidic amino acids, and also of cystine and its oxidation products. Under certain conditions, cystine gives two spots on phenol chromatograms. These spots have equal electrophoretic mobilities. The following findings for the orthocortex (I), para-cortex (II), and cuticle (III) of wool are in agreement with previous results obtained by two-dimensional paper chromatography— (*a*) II contains more arginine and more cystine than does I; (*b*) III is rich in glycine and serine; the cystine spot is absent, and cysteic acid and intermediate oxidation products are present. J.C.F.

Distribution of Cystine between Ortho- and Para-cortex of Wool

M. Leveau

Bull. Inst. Text. France, (74), 75–81 (May 1958)

Wool fibres, hydrolysed with 6 N-HCl at 60°C. for various times from 30 to 120 min., were fractionated into ortho-cortex, para-cortex, and cuticle, and total S and cystine S determined on these fractions. Extrapolation to zero time of hydrolysis indicates that the cystine contents of the ortho- and para-cortex are approximately the same, and the difference in reactivity must be due to some other factor. J.C.F.

Dyeing Behaviour of South African Wools

T. P. van Wyk and D. P. Veldsman

S. African Wool Textile Research Inst. Tech. Rept. No. 10 14 pp. (1957): *Chem. Abs.*, **52**, 10585 (25 June 1958)

Study of the variation of absorption of C.I. Acid Blue 150 on a number of S. African merino wools indicates that finer fibres have both a higher rate and higher total absorption than the coarser fibres. The relationship

between fibre diameter and dye absorbed is a straight-line function over the range 17–28 μ. It is possible to use the dye absorption method to determine average fibre diameter within 1·0 μ. where no fibre damage has occurred; however, weathering damage may extend downward from the tip for a distance equal to 9 months' growth. C.O.C.

Photochemical Degradation of Polyester Fibres
W. Schefer

Textil-Rund., **13**, 336–346 (June), 396–411 (July 1958)

Unfiltered ultraviolet radiation at 285–310 mμ. has a slow but definite adverse effect on physical properties, but causes no yellowing. Normal glass gives protection against these wavelengths. Shortening of the chain occurs with fall in the H_2SO_4 or *m*-cresol viscosities. Humidity change has little influence, nor have delustrants, dyes, carriers, or residues of acid and oxidants, on the light degradation. The damaged fibre is, however, more sensitive to alkaline treatments. S.R.C.

Aromatic Polyanhydrides— A New Class of Fibre-forming Polymers
A. Conix

J. Polymer Sci., **29**, 343–353 (May 1958)

A number of different polyanhydrides containing aromatic nuclei in the main chain have been prepared. They have high m.p. and good hydrolytic stability. Their preparation and properties are described, and an attempt is made to correlate some physical properties with chemical structure. W.R.M.

PATENTS

Rendering Regenerated Cellulose Flame Resistant
R. S. Robe *USP* 2,805,176

If suitable metal compounds are incorporated into the fibre before it is dried after being regenerated, a flameproof of excellent fastness is obtained. C.O.C.

Increasing the Loftiness or Bulkiness of Acrylonitrile Copolymer Yarns
DuP *USP* 2,805,464

Drawn, crimped fibres of acrylonitrile copolymer are treated in absence of tension or moisture at 300–350°F. until they have a temperature of $\leq$ 300°F. The resultant heat-relaxed fibres are then treated, also in absence of tension, with water at 212–280°F. for several minutes. This yields high bulk fibres which yield fabric of very soft handle. C.O.C.

Yarn Lubricant (III p. 704)
Comparative Mechanical Properties of Cotton Fibres and Primary Flax Cells— Effect of Degradation by Hypochlorites (VII this page)
Present Trends in the Russian Manufacturing, Dyeing, and Printing Industries (VIII p. 718)
Modification of the Dyeing Properties of Wool by Sulphuric Acid (X p. 721)
Thermal Degradation of Cellulosic Materials (X p. 721)
Pilling and its Prevention (X p. 721)
Polyacrylic Derivatives. VI— Homogeneous Saponification of Polyacrylonitrile, Polymethacrylonitrile, and Vinyon N (XIII p. 727)
Colour Stabilisation of Acrylonitrile Polymers (XIII p. 727)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Continuous Scouring of Silk
R. V. En'kova

Tekstil-prom., **18**, 31–34 (April 1958)

Several methods are compared, and the best results are obtained when silk fabric is scoured at 80°c. for 25 min. in a bath containing 3·2 g. soda per litre (continuous pH recording), and then treated in a bath containing 7–8 g. oleic soap per litre for 35 min. G.J.K.

Dry-cleaning Today
R. Hess

Melliand Textilber., **39**, 404–407 (April 1958)

A review of dry-cleaning progress with reference to developments in machinery and the effect of solvent and fibre on soil removal. L.A.T.

Comparative Mechanical Properties of Cotton Fibres and Primary Flax Cells— Effect of Degradation by Hypochlorites
A. Parisot, D. Leiber, and O. Topin

Bull. Inst. Text. France, (71), 7–59 (Dec. 1957)

The samples, of a uniform length for each type of fibre, were cut in half in a specially designed apparatus, and one-half used as controls. The samples were given repeated cycles of (*a*) *mild treatment*— hypochlorite of 0·03° chlorometric at 20°c. for 5 min., followed by rinsing in distilled water and drying; (*b*) *severe treatment*— hypochlorite of 0·3° chlorometric at 60°c. for 5 min., followed by rinsing and drying. The controls were given similar treatments in distilled water only. The D.P.s of the treated samples were determined, and the load–elongation curves plotted, giving (i) breaking load, (ii) tenacity, (iii) elongation at break, and (iv) modulus of elasticity from the tangent at the origin. The scatter of the measurements was greater for (i) and (ii) than for (iii). It was also greater for the original samples and those with slight degradation than for severely degraded samples. The following results were obtained: (A) For both fibres, at any stage of the degradation process, (ii) and (iv) are inversely proportional to the fibre fineness, whilst (iii) is practically independent of the fineness. (B) For both fibres, (ii)–(iv) are correlated with the D.P. of the cellulose. (C) Under the mild treatment (and also for the controls), (ii) and (iii) increase for flax cells, whilst (iv) decreases; for cotton, (ii) is almost constant, (iii) is slightly reduced, and (iv) is slightly increased. This is probably due to the plasticising action of the water, together with slight degradation of the cellulose in the case of cotton, which is chemically more vulnerable. (D) Under the severe treatment, (ii) and (iii) show a considerable decrease for both fibres, whilst (iv) increases, the fibres becoming more rigid and therefore weaker. The relation of these findings to the wearing properties of fabrics is discussed. J.C.F.

Bleaching and Mercerising Cotton–Rayon Blended Fabrics
T. R. Scott

Modern Textiles, **39**, 75–79 and 84 (June 1958)

Results of full-scale trials on the bleaching and mercerisation of 80 × 80 print cloth made from blended cotton–rayon (2:1) yarn are compared with those for a cotton fabric of similar weight, construction, and sizing. The latter was treated by pressure kier-boil, hypochlorite, and clip-stenter mercerisation. The blended fabric was similarly processed and was alternatively bleached by open kier-boil and peroxide, by chlorite, and by peroxide continuously, and was alternatively mercerised by expander roll and by pad–mangle processes. Physical properties before and after each processing stage are reported and show no major differences between cotton and the blended fabric processed by the various methods. Attention is drawn to the necessity for rapid and complete hot rinsing after mercerising cotton–rayon blended fabrics. E.C.

Activated Bleaching with Sodium Chlorite
E. Rüedi

Melliand Textilber, **39**, 520–522 (May 1958)

A review of recent technical developments and a description of the Stoffel process of accelerated bleaching with sodium chlorite. L.A.T.

PATENTS

Polyethyleneglycols as Detergent Assistants
U.S. Secretary of Agriculture *USP* 2,806,001

Addition of polyethyleneglycols to aqueous washing liquors greatly reduces the degree of soil re-deposition. C.O.C.

Scouring Flax or Hemp
Montecatini Società Generale per l'Industria Mineraria e Chimica *BP* 798,593

Fibres after retting are treated in an aqueous solution containing an alkali metal sulphite (preferably Na_2SO_3), a neutral alkali metal phosphate or pyrophosphate (preferably Na pyrophosphate or Na_3PO_4) and an alkali metal hydrosulphite or formaldehyde sulphoxylate, at 90°c. These treated fibres are cream and can be bleached to an excellent white, e.g. by H_2O_2 or NaOCl, by use of an appreciably smaller amount of bleaching agent than would otherwise by necessary. C.O.C.

Continuous Bleaching of Cotton Cloth
Food Machinery & Chemical Corpn. *BP* 798,799

The cloth is impregnated with its own weight of an alkaline solution of a peroxy compound and heated with steam. The solution should be free from silicate and contain $\leqslant 0{\cdot}3\%$ on the weight of the cloth of an alkali metal compound yielding orthophosphate ions, a total in the system of 0·02% of a compound yielding alkaline earth metal ions, and a buffer to keep the solution at pH 9–11. Very satisfactory bleaching is obtained and there is no deposit on the sides of the J box. C.O.C.

Chlorite Bleaching
Deutsche Gold- und Silber-Scheideanstalt *BP* 797,951

Excellent bleaching is obtained by impregnating the goods with an activated chlorite solution at $pH < 7$, and bringing to bleaching temperature, e.g. 95–100°C., by steaming them while they are closely packed and subsequently maintaining this temperature until the desired bleach is obtained. C.O.C.

Bleaching Acrylonitrile Fibres
Chemstrand Corpn. *BP* 797,958

Fibres made from a copolymer of $\leqslant 80\%$ acrylonitrile and $\geqslant 20\%$ of a heterocyclic compound containing a tertiary N atom or N ring or a polymerisable alkenyl group substituted thereon, e.g. 2-methyl-5-vinyl-pyridine, are bleached by treating them first with a 0·05 to 0·5% aqueous solution of a strong mineral acid (sulphuric, nitric, hydrochloric or phosphoric acid) and then with aqueous sodium chlorite (2·5–6·0% $NaClO_2$ on wt. of fibres) at 140–180°F. C.O.C.

Combating Streakiness in Poplin (VIII this page)
New Developments in the Wool Industry (X p. 721)

VIII— DYEING

Present Trends in the Russian Manufacturing, Dyeing, and Printing Industries
C. Weimann
Teintex, **23**, 265–269 (April), 416–428 (June 1958)

A report on progress made in the last two years in fibre manufacture, coloration, and colouring matters. S.R.C.

Colour Reaction for Non-ionic and Amphoteric Soaps
J. E. Lovelock and T. Nash
Nature, **181**, 1263–1264 (3 May 1958)

Colloidal electrolytes are known to be able to change the colour of oppositely charged dyes. Similar effects can be obtained by the action of non-ionic soaps on dyes of either charge. Complete discharge of the colour of certain xanthen dyes can be brought about by non-ionic soaps and by water-soluble organic solvents. The percentage concentrations of substances capable of reducing the absorption of dichlorofluorescein to 50% at 490 mμ. are— glycerol 47, glycol 40, methanol 32, ethanol 21, n-propanol 13, acetone 11·5; polyethylene glycol 0·4, polypropylene glycol 0·1, sphingomyelin 0·03, and lecithin 0·025. The colour-discharging phenomenon is attributed to aggregation of the dye. It is suggested that this observation could be used to develop an analytical method for non-ionic soaps. E.V.T.

Soaping of Fabric after Azo Coupling
S. A. Plaksin and N. A. Gribunina
Tekstil. prom., **18**, 42–44 (May 1958)

Two methods are recommended for the washing of cotton fabric freshly dyed with azoic dyes— (*a*) intense washing with hot water, followed by treatment with hot soap and soda solution; (*b*) brief steaming (10 sec.), followed by treatment with a hot soap and soda solution. Finally, the fabric is rinsed with hot and cold water. G.J.K.

Dyeing of Viscose Staple Fibre with Vat Dyes in Circulating Machines
K. Z. Al'tman
Tekstil. prom., **18**, 58–59 (April 1958)

Recipes for the dyeing of viscose staple fibre with vat dyes (green, yellow, and sky blue), in circulating machines are given. Level-dyeing properties are claimed. G.J.K.

Chemical Reactions with Cellulosic Fibres
J. Wegmann
Textil-Rund., **13**, 323–335 (June 1958)

Modification of the fibre properties and permanent finishing with aldehydes and aldehyde resins are briefly discussed. The mechanisms of the combination of reactive dyes with cellulose are examined together with the chemical constitution of such a dye and its component groups. S.R.C.

Dyeing and Printing with Reactive Dyes
R. Crespo
Investigación e Información Textil, **1**, 41–50 (Jan.–March 1958)

The theory of dyeing with reactive dyes is outlined, and the methods of application of Procion (ICI) and Cibacron (Ciba) dyes are compared. S.R.C.

Application of Reactive Dyes in Package Dyeing
J. Annicq
Melliand Textilber., **39**, 414–416 (April 1958)

Working particulars are given for the application of hot- and cold-dyeing reactive dyes— Procion (ICI) and Cibacron (Ciba)— for the dyeing of yarn in package form. L.A.T.

Fixation of Reactive Dyes on Modified Celluloses
T. L. Dawson
J.S.D.C., **74**, 584–585 (Aug. 1958)

Combating Streakiness in Poplin
M. G. Kachurin
Tekstil. prom., **18**, 44–47 (May 1958)

In order to reduce streakiness in poplin it is advisable to gas the yarn prior to singeing the cloth, to avoid sizing whenever possible, to bleach with H_2O_2 in preference to hypochlorite, to mercerise the fabric after dyeing, using a clip mercerising frame and controlling the degree of tension during mercerisation, to conduct mercerisation with high concn. of caustic soda and at relatively low temp., to avoid excessive calendering, and to dye with solubilised in preference to insoluble vat dyes. G.J.K.

Investigation of the Dyeing of Oxidised Cellulose by Infrared Spectroscopy
I. N. Ermolenko and R. G. Zhbankov
Inzhener.-Fiz. Zhur., Akad. Nauk Beloruss. S.S.S.R., (2), 94–98 (1958):
Chem. Abs., **52**, 12405 (25 July 1958)

This method can be used with advantage, e.g. for studying the dyeing of dicarboxycellulose with Auramine, Methylene Blue and Rhodamine. It is based upon the fact that characteristic bands change, e.g. C:O shows very characteristic bands at 1740 and 1610 cm.$^{-1}$. As dyeing progresses, the intensity of such bands changes. C.O.C.

Reservation of Cellulose Triacetate mixed with Cotton during Vat Dyeing
J. Müller
Melliand Textilber., **39**, 541–544 (May 1958)

Most Indanthren dyes are suitable when applied at 45°C. for 45 min. Heat-setting at 210°C. prior to dyeing favours the reserve of cellulose triacetate and improves its resistance to partial saponification. L.A.T.

Temporary Hair Colouring
M. H. Damels
Drug. & Cosmetic Ind., **82**, 158–159, 243–244 (1958):
Chem. Abs., **52**, 11362 (10 July 1958)

A discussion of modern methods of manufacturing colour rinses. C.O.C.

Dyeing of Protein Fibres with Nitroso Dyes formed on the Fibre
P. I. Mikhaĭlenko
Tekstil. prom., **18**, 34–38 (April 1958)

The use of naphtholsulphonic acids, which have affinity for protein fibres, as intermediates in the formation of metallic complexes of *o*-nitrosonaphthols is put forward mainly on economic grounds. A three-stage dyeing recipe is given. G.J.K.

Dyeing of Wool with Solubilised Vat Dyes
A. K. Purenas, V. V. Brazauskas, and V. V. Klusis
Tekstil. prom., **18**, 36–38 (May 1958)

Recipes for the dyeing of wool (fibre or fabric) to light shades with Indigosols in circulating machines are given, and excellent fastness properties are reported. G.J.K.

Fast-to-washing Dyeing of Wool Yarn
J. F. Gaunt

J.S.D.C., **74**, 569–583 (Aug. 1958)

The dyeing of wool yarn with dyes other than the level-dyeing type has become important only since about 1950, but it is now possible to dye almost all colours fast to the S.D.C. No. 2 Washing Test (*B.S.* 2684) and yet sufficiently level for knitting in solid-shade garments. The requirements of the various trades using fast-dyed yarn are considered, and an outline is given of the developments in dyes and dyeing machines leading to the present-day position. Advances have been made in dyes, dyeing machines, and dyeing methods, and these have been complementary. These aspects are discussed separately, and attention is drawn to complications which have arisen as a result of the adoption of new methods. AUTHOR

Dyeing of Wool–Polyester Unions with Palanil (BASF) Dyes— II
A. Würz

Melliand Textilber., **39**, 530–540 (May 1958)

Some Palanil dyes have been examined, and their distribution between the wool and polyester components during single bath dyeing has been investigated. Certain dyeing assistants lead to improved reserve effects on wool. Generally, staining of the wool component can be corrected by soaping and special reduction–clearing treatments. L.A.T.

1:4-Dihydroxynaphthalene Monoalkyl Ethers in dyeing Vinylon
N. Kuroki, T. Kitao, and K. Konishi

Kôgyô Kagaku Zasshi, **59**, 1056–1061 (1956): *Chem. Abs.*, **52**, 10582 (25 June 1958)

1:4-Dihydroxynaphthalene monoalkyl ethers (Alk = CH_3, C_2H_5 propyl, *iso*propyl and butyl) were applied to Vinylon, Amilan or acetate rayon from an aqueous bath for 1 hr. at 80°C., rinsed and treated in 1·5–2·0% aqueous, $K_2Cr_2O_7$, $K_2Cr_2O_7$–$CuSO_4$, $KMnO_4$, Na perborate, H_2O_2, $FeCl_3$ and NaOH–air to obtain blue dyeings. The dyeings were fastest to washing, light, and abrasion when Alk = CH_3 or C_3H_7. The best oxidising agent was Na palmate. The kind of oxidising agent does not affect the chemical nature of the dyes formed in the fibres but it does influence their physical state. C.O.C.

Constitution and Properties of Basic Dyes on Polyacrylonitrile Fibres
J. Wegmann

Melliand Textilber., **39**, 408–414 (April 1958)

The relationship between the constitution of basic dyes and their light fastness was investigated. It is postulated that the light fastness is a function of the basicity of the dye. Light fastness increases as the dye–fibre bond becomes less polar. L.A.T.

PATENTS

Magnesium Compounds for Reducing Consumption of Hydrosulphite in High Temperature Vat Dyeing
American Cyanamid Co. *USP* 2,803,516

Presence of a substantial amount of Mg ions in the bath much reduces the consumption of hydrosulphite when applying vat dyes at high temperatures. Thus a vat was made up of C.I. Vat Blue 6 (0·5) NaOH (6), $NaNO_3$ (1·5), $MgSO_4 \cdot 7H_2O$ (0·25), water (to make 200). It was brought to 200°F. and Na hydrosulphite (6) added. After 50 min. the vat still contained 2·2 parts of Na hydrosulphite whereas a similar vat containing no Mg had a hydrosulphite content of $< 0{\cdot}7$ after only 20 min. C.O.C.

Dyeing Patterns on Warps
Mohasco Industries *USP* 2,804,764

The warp is passed in open width under a series of spray nozzles each of which is individually controlled. The same or different dye liquor is supplied to each nozzle. The warp is then passed under a dryer, e.g. infra-red lamps, to dry and set the dye. C.O.C.

Dyeing with Pigments
FBy *BP* 798,437

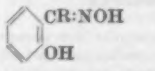
Fabric is padded with a mixture comprising (1) a pigment, (2) an aqueous emulsion of (*a*) a copolymer of a vinyl monomer and vinyl chloride, acrylonitrile, styrene or *as*-dichloroethylene and (*b*) 0–50% on the weight of (*a*) of a vinyl monomer capable of acting as a cross-linking agent, (3) an aqueous solution of high mol. wt. polyacrylamide or an acrylamide copolymer, (4) a urea–formaldehyde, a dicyandiamide–formaldehyde, melamine–formaldehyde or phenol–formaldehyde water-soluble precondensate, and, if desired, (5) an acid catalyst. The mixture must contain no thickener having a basis of volatile solvents. Finally the impregnated fabric is heated to fix the mixture. Thus cotton cloth is padded with a mixture of (1) 40% aqueous emulsion of a copolymer of styrene, butadiene, and acrylonitrile (70 parts by wt.), (2) 10% aq. polyacrylamide (30), (3) urea–formaldehyde precondensate (15), (4) 30% aq. dispersion of Cu phthalocyanine (C.I. Pigment Blue 15), (5) secondary ammonium phosphates (40) and (6) water (to bring to 1000). The padded cloth is dried at 60–80°C. and baked for 4 min. at 135°C. This yields a very level, clear blue dyeing of very good fastnes to washing and rubbing. C.O.C.

Dyeing Polyester and Cellulose Triacetate Fibres
DuP *BP* 797,646

Dyeing at 100°C. with disperse dyes using as carrier a 1:3 to 3:1 mixture by weight of dimethyl terephthalate and benzanilide, there being 2 parts of carrier to each 1000 parts of dyebath by weight, yields level, heavy dyeings of good light fastness and without any spotting or blotchiness. C.O.C.

Colouring Polyester Fibres
ICI *BP* 799,742

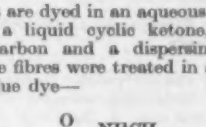
The fibres are treated with a copperable dye and then with an aqueous liquor containing a copper salt and a co-ordination compound of copper with an oxime of formula—



(R = H, Alk, Ar, aralkyl or *cyclo*alkyl), e.g. the copper derivative of salicylaldoxime. The resulting dyeings or prints have excellent fastness to washing, light and dry heat, the fastness to light being superior to that obtained by use of the copper coordination compound in absence of a copper salt. C.O.C.

Dyeing Vinyl Polymers
Ciba *BP* 799,374

Vinyl polymers are dyed in an aqueous bath containing a disperse dye, a liquid cyclic ketone, a halogenated aromatic hydrocarbon and a dispersing agent. Thus polyvinyl chloride fibres were treated in an aqueous bath containing the blue dye—



1:1:3-trimethyl-*cyclo*hexene-(3)-one-(5), chlorobenzene and a dispersing agent. Dyeing commenced cold, the temperature being slowly raised to 55°C. Kept there for 90 min. This yielded a strong, level, blue dyeing. C.O.C.

Dyeing Behaviour of South African Wools (VI p. 716)
Modification of the Dyeing Properties of Wool by Sulphuric Acid (X p. 721)
New Developments in the Wool Industry (X p. 721)
Determination of Dye Selectivity (XIV p. 730)

IX— PRINTING

Chitosan in Pigment Printing
F. I. Sadov and E. O. Vil'dt

Tekstil. prom., **18**, 38–40 (April 1958)

C. Weimann

Teintex, **23**, 558–564 (Aug. 1958)

Chitosan, obtained by hydrolysis of the acetamido groups in chitin—

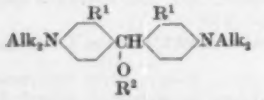
$$C_{32}H_{54}O_{21}N_4 + 2H_2O \longrightarrow C_{28}H_{50}O_{19}N_4 + 2CH_3{\cdot}COOH$$

is used for the preparation of phthalocyanine pigments, acting as both thickening and pigment-building agent. These printing colours are simple to prepare, keep indefinitely, and yield prints on cotton which are fast to washing and rubbing. A recipe for the preparation of chitosan is given. G.J.K.

Chemical Problems in Colour Photography
A. van Dormael
Rev. Questions sci (Belg.–France), **19**, 191–220 (1958): *Chem. Abs.*, **52**, 11635 (25 July 1958)
Review; 28 references. C.O.C.

PATENTS

Spirit-reproducing Carbon Papers
Caribonum *BP* 798,180
A transfer coating is used containing an alkaline substance and a lactone or lower alkyl ether of Methyl Violet 10B (C.I. Basic Violet 3) and Malachite Green A (C.I. Basic Green 4) or a compound of formula—

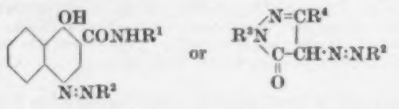


(R^1 = H or CH_3; R^2 = H or Alk). This coating is transferred to a master copy sheet by writing upon the master copy to produce a colourless mirror-script copy on it. The amount of coating transferred is sufficient when the dye derivative is converted to the dye by reaction with an acid substance caused by successively contacting the master copy sheet with a number of spirit-moistened copy sheets, to produce a legible record on each of the copy sheets. C.O.C.

Transfers for Ceramics
Commercial Decal *BP* 797,902
A lithographic transfer for ceramics which is resistant to chemical and mechanical attack when the ceramics are subjected to hot detergent liquids and mechanical agitation, consists of a paper backing, a water soluble gum layer and a layer of pigment containing powdered glass to act as a bonding agent. This pigment layer has a top coating of powdered low melting point glass, the whole being covered with varnish or lacquer. This lacquer protects the transfer but is volatilised when heat is applied to it. During transfer of water-mount transfers it protects the powdered glass from water. C.O.C.

Light Sensitive Diazotype Compositions containing Alumina Pigments
General Aniline *USP* 2,807,544
Image density can be greatly improved by dispersing in the aqueous sensitising solution a chemically pure alumina of particle size $> 5\mu$., e.g. Alon (Godfrey L. Cabot Co.) C.O.C.

Photographic Colour Correction using Coloured and Colourless Colour Couplers
Eastman Kodak Co. *USP* 2,808,329
Coloured colour couplers of formula—



(R^1, R^3 and R^4 = simple or complex Alk or Ar or combinations of these containing one or more solubilising groups; R^2 = a ballasting group), e.g. the yellow 1-*p*-sulphophenyl-3-methyl-4-*p*-laurylphenylazo-5-pyrazolone, which yield dyes which become soluble in the exposed area on development and so can be completely washed out, are used in combination with colourless colour couplers which yield insoluble dyes. The use of the combination results is sharply defined dye images and excellent masking out of undesirable wavelengths. In negative development a picture image is obtained from the coloured coupler and a negative one from the colourless coupler. This results in a colour correction mask with a normal dye image. C.O.C.

Colour Correction in obtaining Accurate Multi-colour Photographs
DuP *USP* 2,803,543

Gaseous Development of Exposed Diazotypes
General Aniline *BP* 797,935

Ultraviolet-absorbing Agents for Use in Colour Photography (III p. 706)
Formation of Azine Dyes in Colour Development. II— Structure of the Azine Dyes from the Arylsulphonyl Derivatives of *m*-Diamines of the Benzene Series (IV p. 710)
Present Trends in the Russian Manufacturing, Dyeing, and Printing Industries (VIII p. 718)
Dyeing and Printing with Reactive Dyes (VIII p. 718)
Colouring Polyester Fibres (VIII p. 719)
Treating Polyethylene Film to render it more receptive of Printing Inks (XIII p. 727)

X— SIZING AND FINISHING

Systematic Study of Sizing. VII— Effect of Yarn Tension in Sizing
R. Schutz and S. Marguier
Bull. Inst. Text. France, (72) 25–39 (Feb. 1958)
The tension applied to cotton yarn during the drying and beaming stages of the sizing operation affects both the dynamometric properties and the abrasion resistance of the yarns. It is recommended that a tension of 30 g. per yarn should not be exceeded. J.C.F.

Mercerisation of Knitted Cotton Fabrics under Tension
H. M. Ulrich
Melliand Textilber., **39**, 417–424 (April 1958)
A specially constructed tensioning device has been employed in mercerising experiments on plain knitted fabrics in 6° and 30°-Bé. NaOH soln. Dimensional changes as a result of mercerising and subsequent treatments were followed by width and length measurements of the knitted loops. The effect of mercerising on lustre and dye uptake was also considered. L.A.T.

Cotton–Rayon Blends for Resin-treated Fabrics
G. V. Lund
Modern Textiles, **39**, 69–75 (June 1958)
The advantages in economy, uniformity, lustre, ease of dyeing and bleaching, and handle of cotton–rayon fabrics are noted together with the limitations of low wet and dry tensile strength and greater shrinkage. The greater sensitivity of cotton to damage by resin treatment and subsequent chlorine retention favours the use of cotton–rayon blended fabrics for resin treatment. The physical properties (tensile and tear strengths and flex abrasion resistance each in the wet and dry condition) of 80 × 80 print cloth made up from cotton, rayon, and blends thereof are reported for the desized and bleached fabrics and also after treatment with Aerotex Resins EU and MW at various levels. At a given resin content rayon gives lower crease resistance but better dry tensile strength than cotton, with blended fabrics intermediate in behaviour. The wet tensile strength after resin treatment does not vary greatly between cotton, rayon, and the blended fabrics, in contrast to the behaviour of the untreated fabrics. Typically, 6·5% resin on a cotton–rayon (75 : 25) blended fabric gives equivalent crease resistance to 5% resin on cotton, and the blended fabric has better dry tensile strength, slightly higher water absorption, and very slightly lower wet tensile and tear strength. Drip-dry rating (appearance after 9 launderings) shows close correlation with crease resistance, and correlation of the latter with dry tensile strength after 14 launderings confirms the advantages of the blended fabrics containing 25–35% rayon. E.C.

Persistence of Aldrin and Dieldrin Residues in Sheep Fleece
I. R. Harrison and C. A. Johnson
Nature, **181**, 1267–1268 (3 May 1958)
The rate of depletion of Aldrin in the fleece has been measured chemically and biologically. The amount falls from 0·2% to 0·05% in four weeks, to 0·025% in nine weeks, and to 0·02% in sixteen weeks. Protection against blowfly strike lasts for at least twelve weeks. Residual Aldrin cannot be extracted by hot solvents or by washing, but it is still able to kill larvae. It is suggested that cloth made from such wool will still be mothproof. E.V.T.

Raising of Wools
B. Monteiro
Tint. ind., (38), 101–127 (April 1958)
The factors affecting the process— raw material, yarn, fabric structure, previous history, moisture content,

pH, and lubricants—are set out, and raising machines are described and illustrated. Fancy effects and defects in the process are included. S.R.C.

Modification of the Dyeing Properties of Wool by Sulphuric Acid
M. Van Overbèke, P. Desprets, and G. Mazingue
Bull. Inst. Text. France, (74), 37–46 (May 1958)

Impregnation of wool fabric in the cold (1 min.) with different concn. of sulphuric acid, followed by—(*a*) drying, (*b*) drying and carbonising at 90°C. for 1 hr., (*c*) drying and carbonising at 120°C. for 1 hr.—and dyeing with Neolan Red GRE (C.I. Acid Red 183), shows that no resist develops until the acid concn. reaches 50° Bé. Uneven dyeing following carbonising must therefore be due to local concn. of acid in the fabric, arising through uneven treatment, e.g. at the nip. In a second series of experiments, the acid concn. was maintained const. at 2° Bé., and the temp. of impregnation varied from 20° to 100°C. (1 hr.). No resist was produced by any of the treatments (*a*)–(*c*). A slight increase in dyeing affinity (up to 5%) as the temp. of impregnation rises is probably due to chain breakdown. When the time of impregnation with 2°-Bé. acid was varied from 15 to 90 min. in the cold, none of treatments (*a*)–(*c*) produced any resist. Impregnation with 66°-Bé. acid for times varying from 15 to 90 sec. produced a strong resist with all three (*a*)–(*c*), the degree of resist increasing directly with the time of impregnation. The least resist was produced by treatment (*c*), and it is suggested that the blocking of amino groups by sulphuric acid is reversible at higher temperatures. J.C.F.

New Developments in the Wool Industry
E. G. H. Carter
Textil-Rund., 13, 383–395 (July 1958)

Modern trends in manufacture and finishing are broadly considered, including developments in carbonising, dyeing, shrinkproofing, mothproofing, quality control, easy-care, and raw-material standardisation. S.R.C.

Finishing of Textiles with Preserving and Proofing Agents and their Testing against Cellulose-attacking Micro-organisms. (A Note on the DIN Proposals No. 53930 and 53933)
W. Hausam and R. Rupp
Melliand Textilber., 39, 429–433 (April 1958)

Good protection against soil decay bacteria and mildew was achieved by treatment with 1·5 and 2·0% (on the weight of material) of dichlorodihydroxydiphenylmethane, Raluben (Raschig) + pentachlorophenol (1:1), and Tetrol (Raschig) + pentachlorophenol (2:1). L.A.T.

Thermal Degradation of Cellulosic Materials
S. L. Madorsky, V. E. Hart, and S. Straus
Bur. Stand. J. Res., 60, 343–349 (April 1958); *Research Paper* 2853

Fortisan, cellulose triacetate, and oxidised cellulose were pyrolysed in a vacuum at 180–465°C. Cotton cellulose and cellulose triacetate were pyrolysed in nitrogen at atmospheric pressure. Tar yields were in decreasing order—cotton > Fortisan > triacetate > oxidised cellulose. Other volatiles consisted mainly of water and carbon monoxide and dioxide from the celluloses, and acetic acid and carbon monoxide and dioxide from the triacetate. Pyrolysis in nitrogen gave less tar than pyrolysis in a vacuum. The tar from the triacetate contained a compound with an infrared spectrum similar to that of the original triacetate. Cotton, Fortisan, and the triacetate do not differ much in their initial rates and activation energies of thermal degredation. Oxidised cellulose has very high initial rates. W.R.M.

Pilling and its Prevention
T. Belleli
Bull. Inst. Text. France, (74), 7–36 (May 1958)

Methods of reducing the pilling tendency of fabrics containing synthetic fibres are based on the reduction of fibre travel, usually by increasing interfibre friction. An instrument for the laboratory study of pilling is described in which samples of the fabric to be tested are rubbed against a Perspex disc coated with a polyurethan foam. Plots of No. of pills per sq. cm. against No. of rubbing cycles are given to illustrate the effect of increasing fibre length and diameter, increasing degree of crimp, and unshrinkability treatments of wool components on the degree of pilling. Steaming of wool–Tergal blends markedly decreases the tendency to pill. The frictional properties of Tergal fibres taken from the blend before and after treatment can be directly correlated with pilling behaviour. Electron microscopy shows considerable modification of the fibre surface. Treatment of Orlon and wool–Orlon blends with a solution of Castile soap (5 g./litre, 15 min. at 40°C.; six immersions, each followed by drying at 20°C.) reduces pilling considerably without impairing the handle of the fabric. Electron micrographs show deposition of soap micelles on the fibres with consequent modification of surface properties. The effect of this treatment on Orlon is compared with impregnation with—(*a*) methoxymethylated nylon, (*b*) glycerol, (*c*) rubber latex, and (*d*) degreasing by alcohol extraction. The soap treatment is less effective than (*a*) and (*b*), but more effective than (*c*) and (*d*). J.C.F.

PATENTS

Chemically Setting the Crimp in Acrylic Fibres
DuP *USP* 2,808,311

The crimped material is treated with a 0·5–40·0% aqueous solution of a compound of formula—

$$R(OH)_m(NH_2)_nA_p$$

(R = benzene or naphthalene ring; m, n and p = 0, 1 or 2; $m + n = 1$ or 2; A = CH_3 or C_2H_5), e.g. phenol or β-naphthol, until shrunk. C.O.C.

Resin Finish having no tendency to Absorb Chlorine
Monsanto *USP* 2,804,402

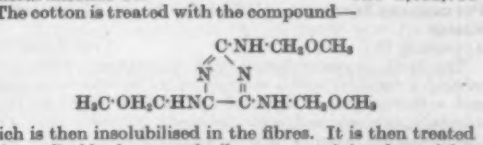
Cellulose textiles are impregnated with an aqueous solution containing (1) a fully methylolated heterocyclic compound having a saturated ring consisting only of C atoms and at least two N atoms to which are attached only one H atom and (2) a thermosetting, infinitely dilutable methyl ether of a polymethylol melamine, e.g. a mixture of a methyl ether of a polymethylol melamine containing 3·4 mol. CHO and 2·2 mol. methanol per mol. of melamine and dimethylol ethylene urea. The ratio of (1) to (2) by weight is 4:1 to 1:2. C.O.C.

Crease-recovery Finish having a Soft Handle
Dan River Mills *USP* 2,808,341

Textiles woven from warps sized with starch or starch derivatives are impregnated with a resinous precondensate, catalyst and amylase simultaneously, kept long enough for the starch to be hydrolysed to a point short of sugar, dried by heat to stop enzyme activity, the resin cure, and the products of starch hydrolysis fixed so that they are fast to washing. C.O.C.

Water-repellent Finish on Cotton
General Electric Co. *USP* 2,802,754

The cotton is treated with the compound—



which is then insolubilised in the fibres. It is then treated with an alkyl hydrogen polysiloxane containing, by weight, 0–200% of an alkyl polysiloxane containing no silicone-bonded hydrogen. Finally, it is baked at > 100°C. to insolubilise the polysiloxane. This yields a water-repellent finish of outstanding resistance to repeated laundering. C.O.C.

Increasing the Strength, Flex Life and Air Permeability of Regenerated Cellulose Cloths
Goodyear Tire & Rubber Co. *USP* 2,809,089

The cloth is treated with < 0·1 N-caustic lye in absence of tension and then "steam-blasted", i.e. the moisture in it is instantaneously converted into steam by passing the cloth between two heated surfaces, the distance between which is less than the original thickness of the cloth, e.g. between two hot calender rolls. C.O.C.

Silicone Finish for Organic Fibres
Dow Corning Corpn. *USP* 2,807,601

A mixture of (1) 5–75% by wt. of a hydrocarbon soluble polysiloxane having 0·05–1·0 Si-bonded H atoms per Si atom, the remainder of the valences being satisfied by O atoms, Alk of < 6 C and > 0·1 Ar radical per Si atom, there being 1·6–2·5 total hydrocarbon radicals and H atoms per Si atom and (2) 95–25% of a hydroxylated polysiloxane of viscosity < 1,000,000 cs. and containing 1·4–2·0 monovalent hydrocarbon radicals per Si atom,

applied to organic fibres and baked yields a water-repellent finish, prevents them being stained by grease or solvents and renders wool fabrics dimensionally stable during laundering. C.O.C.

Water-repellent Finish
Union Carbide Corpn. *BP* 799,808

The material is treated with a linear polysiloxane of formula $(R_3SiO)_z((CH_3)_2SiO)_x(H_2SiO)_y(SiR_3)_2$ (R = monovalent hydrocarbon; $x = 5$–1000; $y = 2$–500; $z = 0$ or 1) in a liquid carrier, preferably using a soap of Zr, Pb, Zn, Co or Fe as catalyst, e.g. Zn octoate, and then cured. C.O.C.

Imparting Flame Resistance to Cellulosic Materials
Dow Chemical Co. *USP* 2,803,562

The material is impregnated with a bis(2-halogenoalkyl)alkenyl phosphonate, preferably in a volatile solvent, e.g. bis(2-chloroethyl)vinyl phosphonate in *o*-dichlorobenzene or ethanol. The handle of the material remains unaffected. C.O.C.

Flame- and Glow-resistant Finish
Bleachers' Assocn. *BP* 799,606

The materials are impregnated with an insoluble resin formed from HCHO and a phosphorodiamidic ester. Thus phenyl phosphorodiamidate (17 g.), 38% formalin (18 ml.) and K_2CO_3 (1·25 g.) were formed into a solution on a water bath. After cooling, the solution was added to urea (17 g.), 38% formalin (34 ml.) and 0·88 ammonia (3 ml.) and stood for 4 hr. $(NH_4)H_2PO_4$ (5 g.) dissolved in water was then added and the whole made up to 250 ml. with water. Spun viscose fabric impregnated with its own weight of this solution, was dried, baked for 3 min. at 145°c., soaped, rinsed and dried. The treated fabric had a full handle, good recovery from creasing and did not readily ignite when treated with a naked flame. Flame was not readily propagated by the fabric and there was no afterglow. The finish had good fastness to washing. C.O.C.

Treating Textiles with Diethylene Glycol Dicarbamate–Formaldehyde Resins
Rohm & Haas Co. *USP* 2,803,563

Textiles of all kinds are treated with aqueous dispersions or solutions of precondensate of the compound $H_2NCOOCH_2CH_2OCH_2CH_2OCONH_2$ with HCHO and either with or without methanol and then baked. This treatment may be used for various purposes. It is especially useful for imparting to thermoplastic textiles stiffness and handle or for increasing the safe ironing point of nylon. C.O.C.

Permanent Mechanical Effects by Use of Cold-curing Resins
Apponaug Co. *USP* 2,802,752

The cloth is impregnated with an aqueous bath containing a catalyst and a mixture of a thermosetting resin and a thermoplastic material and is then dried to 10% moisture content and cold embossed, pleated, etc. The thermosetting resin is a urea–formaldehyde resin, or a urea–formaldehyde-2-methyl-2:4-pentadiol resin and forms 3–15% on the weight of the cloth. The thermoplastic material forms 1–10% on the weight of the cloth and consists of carboxymethyl cellulose, hydroxyethyl cellulose or polyvinyl alcohol. Suitable catalysts are diammonium phosphate or Catalyst AC4 (Monsanto). C.O.C.

Acid Milling
E. F. Drew & Co. *USP* 2,806,804

A handle similar to that obtained by soap milling is produced by acid milling if the carding lubricant is a mixture of a polyethylene glycol (mol.wt. 325–850) monoester of a higher fatty acid (10–20 parts), ethanolamine (1 to 1) condensate with a higher fatty acid (5–10), unsaturated glyceryl triester of higher fatty acid (5–10), and paraffin (67–75). This lubricant resists acid, e.g. in carbonising. Before milling the cloth is treated with an alkoxypolyethylene glycol (mol.wt. 325–850) monoester of a higher fatty acid. If the lubricant has been removed, e.g. by solvent scouring, then in addition to the alkoxypolyethylene glycol ester the cloth must be treated with an acid-stable cushioning or softening agent, e.g. a mixture of polyethylene glycol (mol.wt. 450) mono oleate (44), diethanolamine–coconut fatty acid condensate (22), mustard seed oil (22) and oleic acid (12). This enables a milled finish to be obtained in only three steps, e.g. carbonising, crushing and acid milling, scouring and rinsing. Goods so treated are exceptionally level dyeing. C.O.C.

Rendering Wool Unshrinkable and Non-felting
Director General of the Ministry of Agriculture of Israel *BP* 798,236

Wool is rendered unshrinkable (i.e. resists shrinkage on washing) and non-felting (i.e. resistant to severe milling) by treating it in an aqueous acid bromate solution containing 1·45–6·25% (on the dry weight of wool) of bromate ions, 20–120% of an alkali or alkaline earth chloride (calculated as chloride ion) and at least an equivalent to the bromate ions of a strong mineral acid. Treatment continues until $< 1{\cdot}3$ and $> 2{\cdot}1\%$ in the case where the initial solution contains 2·1% of bromate ions have disappeared from the solution. C.O.C.

Accelerating the Action of Alkaline Thioglycollic Acid on Hair
P. F. A. Grein *BP* 799,432

The contact period needed for aqueous alkaline thioglycollic acid to soften hair is much reduced by adding propyl or *iso*propyl alcohol to the acid. C.O.C.

Mothproof and Water-repellent Finish
Atlantic Refining Co. *USP* 2,804,391

An emulsion type mothproofing composition containing an anionic emulsifying agent, particularly an amine salt of an alkyl aryl sulphonic acid, may be applied to wool cloth without harming its water-repellent properties. Wool cloth may be simultaneously mothproofed and rendered water-repellent by treating it with a mixture of such a mothproofing emulsion and an emulsion of a water-repellent agent. C.O.C.

Light Weight Cellulose Acetate Cloths having Good Resistance to Development of Differential Lustre Effects
BrC *BP* 799,358

Wet spun cellulose acetate yarn of total denier < 100 and filament denier < 5 is woven into a lightweight fabric and then treated to increase the acetyl value of the cellulose acetate without causing appreciable swelling, e.g. by treating with acetic anhydride and a catalyst in an inert solvent, e.g. toluene or CCl_4. C.O.C.

Setting Cellulose Triacetate Fabrics
BrC *BP* 797,662

Modification of *BP* 758,442 (J.S.D.C., **72**, 610 (1956)), formation of wrinkles being avoided by thoroughly wetting the fabric before it is treated with wet steam or water at 120–125°c. C.O.C.

Setting Cellulose Triester Textiles in a Three-dimensional Form
BrC *BP* 797,661

If the processes described in *BP* 753,461, 758,442 and 776,346 (J.S.D.C., **72**, 502,610 (1956); **73**, 529 (1957)) which cause increase in the degree of crystallinity of the cellulose triester are applied to textiles containing cellulose triacetate fibres while they are held in three dimensional form they become set in that form. Thus knitted garments made of cellulose triacetate were wetted with cold water, placed on a former and steamed for 1 min. at 40 lb./sq.in. They received a useful degree of three-dimensional set which was not seriously reduced by washing with water at 80°c. C.O.C.

Knitted Fabrics resembling Suède Fabrics or Buckskin
M. J. A. Laval *USP* 2,805,463

Fabric knitted from yarn consisting of shrinkable polyvinyl fibres blended with non-shrinkable fibres is subjected to a number of shrinking operations firstly while the fabric is wet and then while it is dry. In between the wet and dry shrinkings other finishing operations, e.g. waterproofing, raising, suèding, dyeing, etc., are carried out. Thus such a fabric is rapidly shrunk 50–75% of the total shrinkage required by treating it in open width in water at 90–100°c. A second shrinking of 10–20% of the total required is then given by rotating it while batched on a roller in water at 90–100°c. for 1–2 hr. It is then dyed and given any desired finish. Finally it is dried and treated in open width at 110–140°c. to bring about the remainder of the shrinkage. C.O.C.

Stretchable Fabric
Grove Silk Co. *USP* 2,807,073

Polyamide or polyester yarn which has been set at high temperature is given a twist of 50 t.p.i. and is then set while twisted at a temperature below that of the initial set. It can then be brought to a non-lively or "dead" condition by partly untwisting it, say by 13 t.p.i., and such yarn when heated tends to untwist further, e.g. by 12 t.p.i. The actual amount of this latter untwisting or resumed liveliness is dependent upon the temperature of the successive heat treatments. This principle is used to produce stretchable fabrics by twisting and setting again, untwisting it until the yarn is non-lively, making it up into fabric and then heating to develop further liveliness in the yarn. C.O.C.

Fabric having a Coiled or Cockled Pile
Mohasco Industries *USP* 2,807,072

Thermoplastic yarn which has been coiled by the process described in *USP* 2,662,558 is pulled straight, sized so as to prevent it coiling, woven into pile fabric and then treated with wet steam at atmospheric pressure or with warm water to soften the size and allow the pile yarn to resume its normal coiled form. C.O.C.

Heat Treatment of Glass Fibre Fabric coated with a Thermoplastic
Soule Mill *USP* 2,804,120

The fabric is fed over a system of rolls which impart tension to it and direct it to a clip stenter some distance away from the rolls. Before it reaches the stenter the edges of the fabric are heated before being gripped by the stenter clips. This overcomes the difficulty that the clips prevent the heat reaching the portions of the fabric they grip when the fabric is heated while in the stenter. C.O.C.

Curling Animal Hair with Sulphur Dioxide in Aqueous Solution
Armour & Co. *USP* 2,806,762

Pig, cow or horse hair is spontaneously curled without any need for mechanical curling by treating it with a concentrated aqueous solution of SO_2 at $< 80°C.$, e.g. at 95°c. for 8–15 min. Control of the degree of curl is more precise than in the case of mechanical curling. C.O.C.

Chemistry of Condensation of Commercial Urea–Formaldehyde Resins (III p. 704)
Yarn Lubricant (III p. 704)
Acetylene Diurea–Formaldehyde Resins for Finishing Textiles (III p. 705)
Polyvinyl Chloride Pastes for Coating Textiles (III p. 705)
Bleaching and Mercerising Cotton–Rayon Blended Fabrics (VII p. 717)
Chemical Reactions with Cellulosic Fibres (VIII p. 718)
Combating Streakiness in Poplin (VIII p. 718)
Thermal Degradation of Cellulosic Materials (X p. 721)
Accelerating the Maturing of Linoleum (XIII p. 727)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Stabilisation of Fibre Suspensions
A. J. de Roos

T.A.P.P.I., **41**, 354–358 (July 1958)

The tendency to flocculate of a 0·2% suspension of bleached, unbeaten sulphite pulp in water, and the dispersion obtained by adding cellulose derivatives, were studied by an optical method developed by Robertson and Mason (*Pulp and Paper Mag. Canada*, **55**, 263 (1954)). Methylcellulose, ethyl-hydroxyethylcellulose, sodium carboxymethyl cellulose at $pH > 5$, locust-bean gum, and guar gum are good dispersing agents. Tamarind gum, pectins of low and of high mol.wt., alginate, and starch ether disperse less well; and konnyaku, mesquite, gum arabic, dextrin, dextran, and carragheenin do not disperse at all. Most probably unbranched or but slightly branched high-polymeric molecules with 1-4 glucosidic links are the best dispersing agents. $Al(OH)_3$ and $Ca(OH)_2$ act as dispersing agents because of the increased charge density. R.A.

Paper-coating Trends in the United States
E. J. Heiser

Svensk Papperstidning, **61**, 389–398 (30 June 1958)

Clay accounts for approx. 90% of the paper pigments used in the U.S.A., most of it mined in Georgia and North Carolina, though some English china clay is imported because of its brightness and blue colour. Calcium carbonate and titanium oxide rank next, followed by calcium sulphate, calcium sulphite, barium sulphate, satin white, talc, and zinc pigments, which all generally constitute only a minor portion of the pigment system. This differs from European practice, where satin white and barium sulphate (blanc fixe) are used as major components. Casein is still the most commonly used binder, though starch, protein, and latex binders are increasingly important. 100%-latex binders have been used for boxboard coating. Various coating methods are described and illustrated, and coating formulations given. R.A.

Effect of Alkaline Treatment on Cellulose: Accessibility of Cellulose—III
O. Ant-Wuorinen and A. Visapää

Paperi ja Puu, **40**, 313–321 (June 1958)

The effect of NaOH (0·001–5·0 N.) on cotton linters, bleached sulphite pulp, and viscose rayon at 25°c. and at b.p. was studied by determining the carboxyl and carbonyl group contents, solubility during treatment, D.P. values, X-ray characteristics, and water-vapour sorption capacity. The decreasing effect on the analytical figures for carboxyl and carbonyl contents of different types of cellulose is explained by assuming that an accessibility decrease takes place as a result of the alkaline treatment. A corresponding effect was observed when cellulose was treated with a powerful swelling agent, e.g. calcium thiocyanate. The dissolving effects of different alkali concentrations at different temperatures vary widely, but the variations have no perceptible effect on viscometric D.P. values. R.A.

Reactivities of Lower Aliphatic Anhydrides toward Hydroxyl Groups of Cellulose
C. J. Malm, L. J. Tanghe, H. M. Herzog, and M. H. Stewart

Ind. Eng. Chem., **50**, 1061–1066 (July 1958)

The esterification of the hydroxyl groups of cellulose with acetic, propionic, *n*-butyric, and *iso*butyric anhydrides was studied, (*a*) in solution, using a partly substituted cellulose ester, and (*b*) in suspension, using cotton linters, in the presence of various catalysts. Rates of esterification were influenced by the catalyst and the method of reaction. Degradation during esterification became more severe in the order in which the anhydrides are mentioned above. More degradation was caused by combined than by free sulphuric acid. W.K.R.

Periodate Oxycellulose reduced with Sodium Borohydride
N.-E. Virkola

Paperi ja Puu, **40**, 367–368 (July 1958)

D.P. values for periodate oxycellulose and nitrate D.P. values for the reduced forms are low and decrease when the degree of oxidation increases. D.P. values for the reduced forms correspond in magnitude with each other within the limits of experimental error, irrespective of degree of oxidation. Both unreduced and periodate-oxidised cotton linters resist acid treatment fairly well, while periodate oxycellulose reduced with $NaBH_4$ is degraded even by N-HCl. If the degraded sample is subsequently again reduced with $NaBH_4$, the D.P. does not change. If periodate oxycellulose is degraded with N-HCl and is subsequently reduced with $NaBH_4$, the D.P. increases to almost the same level as the corresponding periodate oxycellulose, undegraded, but reduced with $NaBH_4$. All D.P. measurements were made in alkaline solution. R.A.

Thermodynamic Properties of Dilute Solutions of Cellulose Derivatives
W. R. Moore and B. M. Tidswell

J. Polymer Sci., **29**, 37–52 (May 1958)

Heats and entropies of dilution of dilute solutions of cellulose acetate, ethylcellulose, and cellulose nitrate, in representative solvents, have been obtained. Heats of dilution for cellulose acetate and ethylcellulose systems are generally endothermal, those for the cellulose nitrate systems being exothermal. Entropies of dilution are less

than those obtained for comparable systems containing less polar and more flexible polymers. The results are tentatively interpreted in terms of solvation of polymer and mixing of solvated polymer and solvent. It is possible that thermodynamic properties of dilute solutions of cellulose derivatives may be qualitatively interpreted in terms of simpler lattice theories if solvation is allowed for. W.R.M.

Carboxymethylcellulose. III—Evaluation of Polydispersity by means of the Characteristic *P–D* Curves

J. Schurz and H. Streitzig

Mh. Chem., **88**, 325–329 (June 1957)

From the flow curves obtained from test samples of carboxymethylcelluloses are derived the characteristic *P–D* curves (where P is the D.P. and D is the average velocity gradient in the flow curve) from which the polydispersities are evaluated. The utility of the method is discussed. H.H.H.

Carboxymethylcellulose. II—Preparation of Sodium Carboxymethylcellulose by Multistage Etherification—Viscosities in Water, Neutral Salt Solution, and 2N. Sodium Hydroxide Solution

E. Hayakawa and Y. Morita

Rept. Govt. Chem. Ind. Research Inst. Tokyo, **53**, 77–116 (ix–xi) (March 1958)

Viscosities in neutral salt solution containing 1% of sodium carboxymethylcellulose (NaCMC) depend on both D.S. and D.P. Viscosity is very low at D.S. $< 0{\cdot}4$ and increases with increasing D.S. The Staudinger constant K_m for NaCMC in 2N-NaOH is given by—

$$-\log K_m = 3{\cdot}44 - 0{\cdot}48 \times \text{D.S.}$$

The intrinsic viscosity $[\eta]$ depends on both D.S. and D.P.—

$$\log [\eta] = 0{\cdot}48\ \text{D.S.} - 3{\cdot}44 + \log \text{D.P.}$$

The relative viscosity of NaCMC in pure water varies with D.S. in a very complicated way. At constant D.P. the relative viscosity increases with D.S. up to a maximum at D.S. ca. 0·5, decreases to a minimum at D.S. ca. 1·0, and then increases rapidly with increase to D.S. The logarithm of the relative viscosity is roughly proportional to D.P. and almost independent of D.S. within the range 0·4–2·0. Equations are given relating specific viscosity to D.S. and D.P. The intrinsic viscosity in 2N-NaOH is a function of the viscosity in pure water. W.R.M.

PATENTS

Slime Control in Paper Manufacture

Metal and Thermit Corpn. *BP* 799,120

Slimes may be effectively controlled by (*a*) feeding an emulsion or a self-emulsifiable concentrate of bis-tributyl tin oxide (I) to the system, (*b*) treating internal surfaces in the system with (I) or (*c*) adding (I) directly to the pulp at a point of rapid flow. 0·03 to 0·04 p.p.m. per day (based on the liquid in the system) is suggested for pulps with 2% solids in the circuit; but amounts as low as 0·01 ppm. are effective. The bis-tributyl tin oxide may be used in conjunction with other chemicals, e.g. phenyl mercuric acetate, trichlorophenols, etc. Practically the whole of (I) added to the system appears in the final paper product. The amount of tin in a treated paper having high bioactivity would be about 0·1%, but a range of 1–200 p.p.m. is envisaged. R.A.

Siliceous Brightening Additive

Columbia-Southern Chemical Corpn.

USP 2,786,758

Finely divided calcium silicate is reacted with aluminium sulphate in an aqueous medium containing calcium sulphate.

Various types of calcium silicate may be used. The method of preparation of the silicate determines its properties and hence the properties of the pigment produced. To ensure the production of the calcium silicate in a highly finely divided state, alkaline metal silicate having the composition $Na_2O(SiO_2)_x$, where x is not less than 2 nor more than 4, is preferred.

A paper of high whiteness and brightness is obtained by adding aluminium sulphate to a paper fibre solution, allowing this to permeate the pores of the slurry and then adding the calcium silicate. R.A.

Urea-Formaldehyde Wet-Strength Resins

Hercules Powder Co. *USP* 2,786,823–6

Cationic modified urea–formaldehyde resins, which are stated to improve the wet-strength of paper by approx. 30%, are prepared by treating an aqueous solution of a cationic urea (0·9 mole)–formaldehyde (1·9–2·1 moles) resin, modified by a water-soluble polyfunctional organic nitrogen base (3–30% by wt. of urea), with a cation, e.g. an alkali metal or ammonium ion.

Any water-soluble alkali metal or ammonium salt whose N. solution has a pH of less than about 8 may be used, but by reason of commercial availability and cheapness it is preferred to use ammonium chloride or nitrate, sodium chloride, nitrate or phosphate, potassium chloride or potassium nitrate. R.A.

Triazine Derivatives as Paper Sizes

American Cyanamid Co. *USP* 2,794,737

Cellulosic materials, including paper, are sized by means of a condensate of an alkylamine with hexahydro-1:3:5-triacrylyl-*s*-triazine. The sizing material is prepared by heating 1 to 2 mols of a long-chain saturated fatty amine with 1 mol of hexahydro-1:3:5-triacrylyl-*s*-triazine, which is prepared according to the method given in *USP* 2,559,835. R.A.

Sized Paper

Monsanto Chemical Co.

USP 2,794,736

An aqueous suspension of wood pulp is mixed with suitable quantities of acrylonitrile or methacrylonitrile to which a small amount of a polymerisation catalyst has been added. The reaction is carried out in the presence of a catalyst, such as one of the alkali metal hydroxides, including preferably caustic soda. Exceptionally hard sized paper is produced by this method. R.A.

Writing Paper

Columbia-Southern Chemical Corpn.

USP 2,786,757

Paper of high brightness and opacity is prepared by adding an alkaline earth metal silicate, e.g. calcium silicate, to a paper pulp dispersion in an acidic material which forms an insoluble salt with an alkaline earth metal. Any type of paper pulp may be used. An especially good product is obtained when sulphuric acid, sulphurous acid or aluminium sulphate is used as the acidic material. R.A.

Treating Paper with Silica Sol

DuP *USP* 2,801,938

Paper of increased strength, decreased brittleness and greater resistance to scuffing is produced by impregnating paper with an aqueous stabilised colloidal solution of silica and then irreversibly attaching the silica to the fibres, e.g. by acid treatment or by heating to 50°C. at a concentration of SiO_2 in the solution of $> 45\%$. C.O.C.

Starch–Clay Coating Colours for Paper

American Cyanamid Co. *USP* 2,804,436

Clay–starch coating colours containing a water-soluble high mol. wt. anionic linear carbon chain polymer, $\leqslant 20\%$ of the C atoms of the chain curing one ionic COOH group each, e.g. a copolymer of acrylic acid and acrylamide, yield more strongly adhesive and durable coatings than those obtained in absence of the polymer. There is only very slight increase in the viscosity of the composition. C.O.C.

Coated Paper

Western Electric Co. *USP* 2,797,171

Thin coats of insulating lacquer are applied to thin strips of paper in a uniform layer. A web of material is advanced continuously over a coating roll which applies the lacquer. A light is positioned on one side of the paper and light-measuring means are reciprocated back and forth across the paper on the other side of the light. Means may be provided to give a constant indication of the amount of light passing through the paper at any position of the light-measuring means.

The coating is said to be held between the limits of 0·12 and 0·20 mg. of lacquer per cm² of paper, giving a thickness of about 0·00005 inch. R.A.

Coated Paper

Rohm & Haas Co. *USP* 2,790,735

The coating consists of a finely divided pigment, mainly

clay, (100 parts) and a salt of a copolymer (8–25 parts) of an alkyl acrylate with 4–7% by weight in the copolymer of acrylic or methacrylic acid. The alkyl acrylate should contain 1–4 carbon atoms in the alkyl group. The copolymer has a Ti value of not over 20°c.

USP 2,790,736

As above but the copolymer contains 2·5–7·0% by weight in the copolymer of at least one of the following acids: itaconic, aconitic, maleic, fumaric, the dimer and the trimer of methacrylic acid. R.A.

Coated Paperboard

Gardner Board & Carton Co. *USP* 2,790,730

The surface of the paper or board is worked by the rolls of the breaker stack of calenders in the presence of a plasticising solution and is thus prepared for the reception of a very thin imprinted coating of mineral-adhesive mixture. The coating is applied before the surface of the web has completely dried.

After having been imprinted, the board is dried and is then usually passed through one or more finishing stacks of calender rolls. R.A.

Photographic Coating Compositions

Gevaert *BP* 799,184

A solution in a volatile solvent, of gelatine and a nitration product of alginic acid or its partial ester produces adhesive compositions which will adhere both to gelatine and to cellulose ester films and which will not dissolve in alkaline baths. The ratio by weight of the nitration product to gelatine should be between 9:1 and 1:9 and it is prepared by reaction of fuming HNO_3 on alginic acid or a metal alginate. By suitable choice of reaction conditions and starting materials the degree of substitution can be controlled. The alginic acid nitrate may contain 0·5–1·5 nitrate groups per mannuronic unit and the carboxyl group of the latter may be esterified with an alcohol or an alkylene oxide. R.A.

Moisture-resistant Paper

Commonwealth Engineering Co. *USP* 2,786,786

A dextran or dextran partial conversion product is incorporated into the paper stock by addition to the beater or by application to the paper at a subsequent stage in the process. The dextran product should contain chemically bound radicals derived from higher fatty acids containing 8 to 18, preferably 12 to 18 carbon atoms. R.A.

Laminated Transfer Device for Manifold Stationery

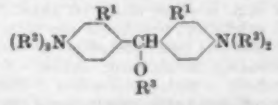
Standard Register Co. *USP* 2,790,742

An absorbent paper is coated with ink, a flow metering permeable wax applied to the underside and an impermeable plastic film applied to the topside. This film is curled transversely to its length and then released immediately prior to contact application to the paper, whereafter the material is passed through rollers to fix the film upon the paper. R.A.

Manifolding

Caribonum *BP* 798,396

Paper is coated on one side with an alkaline substance, e.g. Na_2CO_3, and a colourless compound of formula—



(R^1 = H or CH_3; R^2 = same or different Alk, Ar, alkaryl or aralkyl; R^3 = H or Alk), e.g. 4:4′-(tetraethyldiamino)-diphenylcarbinol. An acid substance which reacts with the above substance to yield a dye is also embodied in a coating which is applied to the paper either on the same side as the first coating or preferably on the other side. A barrier layer containing, e.g. gelatin, is applied to prevent the two layers coming into contact until the barrier layer is broken by the paper being written on. An opaque white pigment may be incorporated in any or all of the layers and an oily substance of refractive index similar to that of the white pigment forms part of the barrier layer. This renders the opaque white pigment transparent on the parts where pressure is applied in writing and so gives enhanced contrast. C.O.C.

Medicated Fibrous Materials

H. Klenk *BP* 799,273

A fibrous carrier, e.g. paper, cellulose, cellulose wadding, etc., is coated with a composition containing one or more active substances, e.g. a polychlorophenol such as 2:2′-dihydroxy-3:5:6:3′:5′:6′-hexachloro-diphenylmethane, together with an alkyl aryl polyglycol ether or a wetting agent derived from a lipophilic or hydrophilic colloid and a paper or textile size. Comminuted or discrete fibrous substances similar to those used in the carrier web and/or a carrier, e.g. kieselguhr, may be added to the coating mixture which is applied in the form of an aqueous suspension or emulsion by any known method, e.g. spraying, dipping or mixing. R.A.

Apparatus for Coating Paper (I p. 703)
Fixation of Reactive Dyes on Modified Celluloses (VIII p. 718)
Determination of the Degree of Polymerisation of Cellulose from Viscosity Data in Benzyltriethylammonium Hydroxide (XIV p. 731)

XII— LEATHER; FURS; OTHER PROTEIN MATERIALS

Electroendosmosis through Hide and Leather Membranes

B. N. Ghosh and S. Ghosh

Bull. Central Leather Research Inst., Madras, 4, 259–265 (1958): *Chem. Abs.*, 52, 12438 (25 July 1958)

The isoelectric points of dehaired, delimed skin and various leathers were measured by an electroendosmotic method. ζ-Potentials at various pH were calculated from the Smoluchowski relation; the pH with ζ-potential zero was taken as the isoelectric point. Values of the isoelectric point for skin and leather tanned with HCOH, vegetable tannin and Cr (the chrome leathers were neutralised with borax to pH 7 before washing free from electrolytes and equilibrating with solutions of various pH) agree with those of previous workers. The lower value of the isoelectric point for chrome leather dried before neutralising, compared with chrome leather that had not been dried, was confirmed. The low isoelectric point of vegetable tanned leather is caused by free acidic groups in the combined tannin. C.O.C.

Production of Corrected Grain Leathers

K. Eitel

Das Leder, 8, 265 (1957): *J. Soc. Leather Trades Chem.*, 42, 219 (June 1958)

Components of typical finishes are outlined and illustrated by photomicrographs of the diluted finishes. Polymer particles are smaller than non-polymer binding materials. Plasticisers for protein binders are divided into gelatinising (e.g. glycerol) and non-gelatinising (e.g. sulphated oils). Formation of the finish film is described; for maximum fastness properties and elasticity it is essential to have sufficient polymer in the finish to embed and surround both pigment and non-resin binder. Finally, a series of photographs shows the appearance of a leather in various stages of finishing for a corrected grain leather. C.J.W.H.

Influence of Glazing on the Properties of Upper Leather

R. Schubert

Das Leder, 8, 241 (Oct. 1957): *J. Soc. Leather Trades Chem.*, 42, 219 (June 1958)

A general discussion on the influence of glazing box calf, aniline calf, chrome side, and glacé kid on their appearance, behaviour towards finishes, waterproofness, and strength properties. C.J.W.H.

Glacé Kid and Glazed Sheep Leathers

G. Walther

Das Leder, 8, 249 (1957): *J. Soc. Leather Trades Chem.*, 42, 219 (June 1958)

A consideration of finishing problems in the manufacture of these leathers. C.J.W.H.

Laboratory Screening Test for Bactericides for Use in Sheepskin Soaking Pits

J. H. Richardson

Appl. Microbiol., 6, 142–145 (1958): *Chem. Abs.*, 52, 12437 (25 July 1958)

The following bactericides reduced the bacterial count > 95% (at the concentration indicated): Iobac 0·40%, Iosan 0·40, $C_6H_5HgO{\cdot}OCCH_3$ 0·05, Na_2SiF_6 0·25%,

$(NH_4)_2$ SiF_6 0·10, $ZnSiF_6$, 0·05, $MgSiF_6$ 0·15, Cu 8-quinolinolate 1·0, *o*-benzyl-*p*-chlorophenol *o*-phenylphenol (Socci 6137) 0·5, 2-benzyl-4-chlorophenol (Socci 6125) 0·5, pyridinethione Na salt 0·0001, 3-cocotetrahydropyrimidine 0·5, 3-soyatetrahydropyrimidine 0·5, 2-methyl-3-cocotetrahydropyrimidine 0·5, 2-methyl-3-soyatetrahydropyrimidine 1·0, 2-methyl-3-tallowtetrahydropyrimidine 1·0, gelatin thiocarbamate pentachlorophenolate 0·5, Armac C 0·5, Duomac C 0·25, Duomac T 0·5, Duomeen T 0·5, Duomac C and Ethofat 242/25 0·2.

C.O.C.

Charge and Deformation of Protein Molecules

A. G. Pasynskii

J. Polymer Sci., **29**, 61–68 (May 1958)

The high rigidity of globular protein molecules is largely due to hydration. The contribution from the charge is much less. In buffered solutions, if hydration is allowed for, the shape of gelatin particles is practically independent of pH, and in unbuffered solutions it varies roughly in proportion to the fourth or fifth root of the number of charges. In the cases of serum and egg albumins the effect of charge is negligible. W.R.M.

Effect of Synthetic Dispersions on the Preservative and Fungicidal Ingredients of Emulsions (Leather Finishes and Paints)

W. Hausam

Leder Häutemarkt tech. Beilage (Dec. 1956): *J. Soc. Leather Trades Chem.*, **42**, 216 (June 1958)

The addition of dispersions of polymers to finishes and paints containing casein or albumen reduces the effect of disinfectants and fungicides. The results of tests are given in which *p*-chloro-*m*-cresol, alone or in combination with pentachlorophenol, protects protein-containing finishes from deterioration. C.J.W.H.

PATENTS

Resins as Tanning Agents

Boehme Fettchemie *BP* 797,536

Excellent tanning is obtained by use of a cationic and an anionic resin either successively or simultaneously or by use of the resin salt obtained by reaction of equimolecular quantities of these resins with one another. C.O.C.

Filling of Leather

ICI *BP* 798,649

Modification of *BP* 585,135 (J.S.D.C., **63**, 194 (1947)). The leather is drummed before, during or after, tanning with an aqueous dispersion of a water-insoluble, solid interpolymer of an acrylic or methacrylic ester with an ethylenically unsaturated, polymerisable carboxylic acid, e.g. an interpolymer of methyl methacrylate and methacrylic acid. C.O.C.

Polyalkylene Oxide Wax Derivatives for Oiling Fur Skins

Boehme Fettchemie *BP* 798,464

Aqueous emulsions containing natural or synthetic oils or fats, polyalkylene oxide wax derivatives containing lipophilic groups and natural or synthetic waxes, are excellent oiling agents for fur skins to which they may be applied at any stage of processing. C.O.C.

Treating Fur Skins

C. R. Carney *USP* 2,807,557

The skins are treated with an oil-in-water emulsion of a tanning agent, e.g. tannic or oleic acid, a hydrocarbon wax, e.g. ozokerite, a silicone resin and a silicone oil. The treated skins can be wetted and dried without hardening, the hairs remain firmly fixed in the skin and stand out and fluff up. They have high gloss or shine and no oiliness. C.O.C.

XIII— RUBBER; RESINS; PLASTICS

Effect of Structure on Thermal Decomposition of Polymers

L. A. Wall and R. E. Florin

Bur. Stand. J. Res., **60**, 451–458 (May 1958) *Research Paper* 2860

Rate data on thermal decomposition are discussed from the viewpoint of depolymerisation as a free-radical chain reaction. Most characteristics of the thermal-decomposition rate curves are results of basic structure and not of trace impurities, although the magnitude of rates and activation energies are sensitive to trace impurities. Intermolecular transfer will account for rate behaviour in a large number of polymers. W.R.M.

Urea–Formaldehyde Condensation

G. Zigeuner and W. Hoselmann

XVI— Reaction of Urethan with Formaldehyde

Mh. Chem., **88**, 5–10 (Feb. 1957)

Urethan and formaldehyde condense in an alkaline medium to give a dimethylene ether—

$$C_2H_5{\cdot}O{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}NH{\cdot}COOC_2H_5$$

and not a hydroxymethylmethylenediurethane—

$$C_2H_5{\cdot}O{\cdot}CO{\cdot}N(CH_2OH){\cdot}CH_2{\cdot}NH{\cdot}COOC_2H_5$$

as previously reported by Einhorn (*Annalen*, **361**, 130 (1908)). Excess formaldehyde reacts with urethan in alkalinic medium to give products which on subsequent treatment with alcohols and acids afford mixtures of acetals.

XVII— Reaction of Urea with Formaldehyde in Butanol

Ibid., 159–160

Fahrenhorst's statement (*Kunststoffe*, **45**, 43 (1955)), that equimol. amounts of urea and formaldehyde react in butanol at 80°c. in the presence of alkali to give no monohydroxymethylurea but an adduct of urea and formaldehyde, is now shown to be incorrect. Repetition of Farenhorst's experiments has afforded large amounts of monohydroxymethylurea—

$$NH_2{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}OH$$

and a bicarbamidomethyl ether—

$$NH_2{\cdot}CO{\cdot}NH{\cdot}CH_2{\cdot}O{\cdot}CH_2{\cdot}NH{\cdot}CO{\cdot}NH_2$$

H.H.H.

Viscometry of Poly-6-caproamide (6-Nylon)

F. Rybnikar

J. Polymer Sci., **29**, 519–530 (May 1958)

The influences of water in the tricresol used as solvent, molecular weight, and temperature are studied. Water reduces the intrinsic viscosity $[\eta]$. The Huggins constant K increases with mol. wt. and is replaced by a function—

$$K = K_1 + K_2/[\eta]^x$$

where $K_1 = 0{\cdot}26$, $K_2 = 285{\cdot}5$, and $x = 1{\cdot}48$. At higher temperatures $[\eta]$ decreases and K remains constant. $[\eta]$ is related to temperature by—

$$\log [\eta] = \frac{a}{T} + b$$

where a is independent of $[\eta]$ and b is a function of $[\eta]$. W.R.M.

Hetero-chain Polyamides. VIII— Solubility of Mixed Polyamides

T. M. Frunze and V. V. Korshak

Izvestiya Akad. Nauk S.S.S.R., otdel khim. nauk, 344–352 (March 1958)

The relation between the solubility and the structure of different mixed polyamides is investigated, and it is shown that the mixed polyamides possess greater solubility than the homogeneous polyamides. The relation between structure and m.p. is more clear-cut than that between structure and solubility: the lower the m.p. the greater the solubility, and the less ordered is the structure. The order of decreasing solubilising action of solvents is phenols > alcohols > methylcellosolve > ethylcellosolve. The presence of aromatic components in the mixed polyamide causes a decrease in the solubility. G.J.K.

Hetero-chain Polyesters

V. V. Korshak, V. A. Zamyatina, and N. I. Bekasova

IX— Catalyst for Polyesterification

Izvestiya Akad. Nauk S.S.S.R., otdel. khim. nauk, 482–485 (April 1958)

The catalytic action of a series of metallic oxides and hydroxides and LiF on the preparation of polyethylene *tere*phthalate is investigated.

X— Kinetics of Polycondensation of Hydroxyethyl *tere*Phthalate

Ibid., 486–491

The kinetics of polyesterification of hydroxyethyl *tere*phthalate at 240–285°c. under atmospheric pressure is investigated, and the energy of activation found to be 35 kcal./mole. Reaction is of the second order irrespective of the effect of the catalyst (LiOH in the presence of

Al_2O_3), the latter increasing the reaction rate by 2–3 times. After heating for 6–7 hr. at 275°C., and for 3 hr. at 285°C., the equilibrium is established, and further heating does not change the mol. wt. of the polyester. G.J.K.

Heat and Ultraviolet Aging of Polyvinyl Chloride
C. F. Bersch, M. R. Harvey, and B. G. Achhammer
Bur. Stand. J. Res., **60**, 481–488 (May 1958)
Research Paper 2863

Four polyvinyl chloride polymers, prepared with different initiators, were exposed to ultraviolet radiation and heat, in a vacuum and in air. The gaseous products were analysed by mass spectrometry. Benzene was among the products evolved in most cases. Susceptibility to degradation increased with increasing oxygen content and unsaturation of the original polymer. A two-stage degradation process is suggested involving dehydrochlorination and decomposition of the resulting polyene chain. Colour formation is attributed to oxidation and conjugated unsaturation. W.R.M.

Polyacrylic Derivatives. VI—Homogeneous Saponification of Polyacrylonitrile, Polymethacrylonitrile, and Vinyon N
H. Stübchen and J. Schurz
Mh. Chem., **89**, 234–238 (May 1958)

The title substances are submitted to homogeneous saponification by sodium hydroxide in dimethylformamide and dimethyl sulphoxide respectively, and the ultraviolet spectra of the reaction products are reported. Polymethacrylonitrile is appreciably more difficult to saponify than polyacrylonitrile. H.H.H.

PATENTS

Treating Polyethylene Film to render it more receptive of Printing Inks
DuP *USP* 2,801,446

The film is treated at 150–325°C. with ozone, preferably under ultraviolet radiation, and is then quenched in aqueous hydrogen peroxide, nitrous acid, alkaline hypochlorite, HNO_3 or a mixture of HNO_3 and H_2SO_4.

USP 2,801,447

The film is treated at 25–95°C. in an aqueous bath containing a halogen, HCl, HBr, HI or HF.

USP 2,805,960

The polyethylene is treated with ozone at 150–325°C. in presence of ultraviolet radiation and then quenched in an aqueous bath containing a halogen or HCl, HBr, HI or HF. C.O.C.

Patent Leather-like Finish on Polyvinyl Chloride Sheeting
DuP *USP* 2,801,949

Polyvinyl chloride sheeting is laminated to a film of dissimilar material by passing the assembly through calender rolls. One of the rolls may be heated or the polyvinyl chloride surface may be heated immediately before the laminating. The bond between the two materials must be such that (*a*) no separation occurs when the laminated product is rolled on a 3 in. diameter core and (*b*) the dissimilar film can be stripped from the polyvinyl chloride without marring the surface finish. The polyvinyl chloride is then moulded against the dissimilar film. When the two are separated the polyvinyl chloride sheeting is as smooth as the dissimilar film and has a mirror-like finish. C.O.C.

Flushing Poly(Vinyl Chloride) Stabilisers
National Lead Co. *BP* 798,675

Dibasic lead salts of fatty acids 6–22 C useful as stabilisers, e.g. for poly(vinyl chloride), are prepared in aqueous media and are flushed directly into water-immiscible organic media, e.g. plasticisers, by agitation therewith.

BP 798,444

Basic lead salts, e.g. dibasic lead phosphite, prepared in aqueous media are flushed into water-immiscible organic media by agitation in presence of 0·5–5·0% of a fatty acid (6–22 C atoms). E.C.

Basic Organic Salts of Cadmium as Stabilisers for Vinyl Halide Resins
National Lead Co. *BP* 799,568

Compounds of formula—

$$CdO\text{-}Cd[R(COO)_{2n}]_n$$

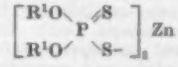
(n = 0·5 or 1·0; R = aliphatic radical of 1–17, an aromatic radical of 6–11 C or, when n = 1, the linking bond of oxalic acid), e.g. monobasic cadmium caprylate, are excellent stabilisers for polyvinyl halide resins. As they are white they can be used in light-coloured and semi-transparent stocks. C.O.C.

Finishing Artificial Leather
A. A. Alegre *BP* 798,063

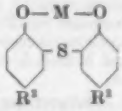
Artificial leather produced by bonding fibres together with rubber latex or a synthetic resin is treated with a solution or paste of acrylic resin, in presence or absence of casein. The treated surface is then coated with polyvinyl butyral, polyvinyl acetate or polyvinyl chloride. Finally the artificial leather is calendered at > 100 kg./sq.cm. The coating composition can contain pigments, fillers, plasticisers, etc. C.O.C.

Colour Stabilisation of Acrylontrile Polymers
American Cyanamid Co. *USP* 2,801,987

Incorporation of zinc phenyldithiocarbamate, or a compound of formula—



(R^1 = Ar, Alk, aralkyl or *cyclo*alkyl), e.g. dimethyldithiophosphate, or a compound of formula—



(M = Ba or Ca; R^2 = Alk of 1·20 C), e.g., Ba octyl phenol sulphide, stabilises acrylonitrile polymers against discoloration on heating. C.O.C.

Permanently Flexible Floor Coverings
National Starch Products *BP* 797,630

A floor covering that can be made on conventional linoleum manufacturing equipment but which does not need the higher temperature—high pressure equipment necessary for making most of the vinyl floor coverings, is obtained by using as the binder for the pigments and fillers a resinous copolymer of vinyl acetate and another monomer which acts to plasticise the resultant copolymer, e.g. dibutyl fumarate or maleate.

BP 798,676

The binder is a dry, granulated terpolymer of vinyl acetate, vinyl chloride and a monomer, e.g. vinyl propionate or vinyl stearate, which acts as an internal plasticiser for the terpolymer after the mass has been sheeted. C.O.C.

Accelerating the Maturing of Linoleum
ICI *BP* 799,869

Incorporating a carboxylic hydrazide, e.g. sebacic dihydrozide, in linoleum compositions reduces the maturing time of linoleum compositions. C.O.C.

Chemistry of Condensation of Commercial Urea–Formaldehyde Resins (III p. 704)

XIV—ANALYSIS; TESTING; APPARATUS

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XIV—3-Bromo- and 3:3′-Dibromo-benzidine as Redox Indicators
R. Belcher, A. J. Nutten, and W. I. Stephen
J.C.S., 2336–2338 (June 1958)

The behaviour of 3-chloro-, 3:3′-dichloro-, 3-bromo, and 3:3′-dibromo-benzidine in conventional inorganic redox systems is described, and an assessment reported of their value as redox indicators. When used in the titration of iron(II) with potassium dichromate and ceric sulphate, all four behave reversibly, but only the bromo and dibromo cpd. afford sharp enough end-points (colourless to yellow) to have practical applications. At elevated temp. the indicator reaction still occurs but is accompanied by some destruction of the reversibly formed oxidation products. Potentiometric data show that both the bromobenzidine indicators function in the potential range over which the end-points of these titrations occur. H.H.H.

Analytical Reviews
Anal. Chem., **30**, 553–872 (April 1958)
L.T.W.

Volumetric Determination of Nitrate Ion. Simultaneous Determination of Nitrite Ion
O. R. Gottlieb and M. T. Magalhaes
Anal. Chem., **30**, 995–997 (May 1958)

The reaction between sulphamic acid and nitrate in presence of conc. H_2SO_4 is used in the determination of nitrate ion, excess sulphamic acid being titrated with sodium nitrite soln. Double titration eliminates the effect of any nitrite in the sample. By an additional titration with the same solutions and techniques, nitrite can be determined simultaneously. L.T.W.

Preparation and Analytical Properties of *NN*-Di-(carboxymethyl)aminomethyl Derivatives of some Hydroxyanthraquinones
R. Belcher, M. A. Leonard, and T. S. West
J.C.S., 2390–2393 (June 1958)

The *NN*-di(carboxymethyl)aminomethyl deriv. prepared from alizarin and Alizarin Bordeaux R by a Mannich-type condensation with formaldehyde and iminodiacetic acid are particularly useful as indicators in complexometric titration, and especially in the ethylenediaminetetra-acetate (E.D.T.A.) titration of Pb^{2+}, Zn^{2+}, Co^{2+}, and In^{3+} in acid soln. are noted. The indicators are stable in soln. for several months, and show promise as possible colorimetric reagents for Th^{4+} and Ce^{3+} since the chelate cpd. formed have an intense colour which is not affected by fluoride and E.D.T.A. to the same extent as the Alizarin S chelates. H.H.H.

Spectrophotometric Determination of Carbonyl Oxygen
F. H. Lohman
Anal. Chem., **30**, 972–974 (May 1958)

An extremely accurate method for the determination of carbonyl oxygen depends on condensation with 2:4-dinitrophenylhydrazine to form highly coloured 2:4-dinitrophenylhydrazones. The hydrazone is selectively extracted with hexane to eliminate reagent interference. The absorbance is measured at 300 mμ., the effective range being 3–300 p.p.m. The method has been applied to soya-bean and cottonseed oils and to laurone in synthetic samples. L.T.W.

Determination of Aromatic Nitro Compounds
W. B. Koniecki and A. L. Linch
Anal. Chem., **30**, 1134–1137 (June 1958)

A direct analytical procedure specific for aromatic nitro compounds is based on the reduction of the nitro group to primary amine by thiourea dioxide in alkaline soln. Diazotisation followed by couplings with Chicago acid (8-amino-1-naphthol-5:7-disulphonic acid) produces a red colour. Excess of the reducing agent is destroyed by heating at 50°c. L.T.W.

Submicro-methods for the Analysis of Organic Compounds. III— Determination of *N*-Methyl Groups and the Simultaneous Determination of Alkoxyl and *N*-Methyl Groups
R. Belcher, M. K. Bhatty, and T. S. West
J.C.S., 2393–2396 (June 1958)

A modification of the Herzig–Meyer method (*Mh. Chem.*, **15**, 613 (1894); **16**, 599 (1895); **18**, 379 (1897) is reported whereby *N*-methyl groups are determined with an absolute accuracy of ± 0·3% on 50-μg. samples. Further, by a slight modification of procedure, methoxyl and *N*-methyl groups may be determined simultaneously on the same sample with equal accuracy. H.H.H.

Standardisation of Test Methods for Textile Auxiliaries— XII
E. Götte
Melliand Textilber., **39**, 561–562 (May 1958)

Olive oil or paraffin oil is dissolved in a volatile solvent and is evenly applied to degreased wool. An oil-soluble dye such as Ceres Violet BRN (FBy) is used as an indicator. After scouring with an agent under investigation, rinsing, and drying, the wool is extracted with an organic solvent, and the fatty matter in the extract is estimated gravimetrically or colorimetrically. The amount of residual oil in relation to the concentration of the scouring agent gives a measure of its scouring efficiency. Results of inter-laboratory trials have shown that this procedure is suitable for olive oil but not for paraffin oil. A colorimetric method is not applicable in the latter case because the ratio of oil to oil-soluble dye does not remain constant as a result of scouring. L.A.T.

Tannins. VII— Colorimetric Determination of D-Catechin in Catechin (C.I. Natural Brown 3)
K. Kimura, Y. Satomi, and S. Kuwano
Yakugaku Zasshi., **78**, 325–329 (1958): *Chem. Abs.*, **52**, 12439 (25 July 1958)

D-Catechin was determined colorimetrically with vanillin–HCl reagent. D-Catechin and gambier tannin were effectively separated by paper chromatography, from which the D-catechin was eluted out by methanol and determined. In this way the sum of D-catechin and of its optical isomers was obtained and expressed as D-catechin. C.O.C.

Chromatography of Dye Intermediates. VIII— Paper Chromatography of 2-Amino-8-naphthol-6-sulphonic Acid and 2-Amino-5-naphthol-7-sulphonic Acid, and Identification of 2:8-Diaminonaphthalene-6-sulphonic Acid
J. Latinák and L. Skalický
Chem. Listy, **52**, 631–635 (1958): *Chem. Abs.*, **52**, 12404 (25 July 1958)

Commercial 2-amino-5-naphthol-7-sulphonic acid (I), and 2-amino-8-naphthol-6-sulphonic acid (II) can be separated on paper impregnated with 5% aq. $NaHCO_3$ in the system propyl alcohol–aq. $NaHCO_3$ (2:1). I contains 2-amino-5:7-naphthalenedisulphonic acid, 2-amino-5-naphthalenesulphonic acid, bis(5-hydroxy-7-sulpho-2-naphthyl)amine, and 2:5-dihydroxy-7-naphthalenesulphonic acid. II contains bis(8-hydroxy-6-sulpho-2-naphthyl)amine, 2-amino-6:8-naphthalenedisulphonic acid, 2:8-dihydroxy-6-naphthalene sulphonic acid (III), 2-amino-6-naphthalene sulphonic acid and 2:8-diamino-6-naphthalenesulphonic acid (IV). IV was identified by mild acid hydrolysis yielding II, III, 2-amino-8-naphthol and 2:8-dihydroxynaphthalene; it was obtained by aminating II at 200°c. and 20 atm. C.O.C.

Paper Chromatography of Some Acid Dyes
J. Kolšek
Chemiker-Ztg., **82**, 35–36 (1958)

Seventeen acid dyes (Acilan) were subjected to paper chromatography using the solvent recommended by Ekman (*Chem. Abs.*, **43**, 2263 (1949)) and an aqueous solution of the dye. A 100-cm. measuring cylinder (20 cm. high) was used as the chamber with a strip of special paper as a sorbent. It is best, especially with mixtures containing very small proportions of some components, to apply the sample across the entire width (6–8 cm.) of the paper. The colours and R_f values of the zones obtained with each dye are given. C.O.C.

Analysis of Hair Dyes— II
C. J. Turi
Rend. Ist. Super. Sanità, **20**, 570–589 (1957): *Chem. Abs.*, **52**, 11352 (10 July 1958)

The dyes are fractionated by (1) acidifying with HCl, extracting with ether, and evaporating the extract (phenols and neutral substances); (2) making the aqueous phase alkaline with dil. NaOH in presence of $NaHSO_3$, extracting with ether and evaporating the extract to near dryness under CO_2 (amines and diamines); (3) acidifying the aqueous phase slightly with HCl, neutralising with excess $NaHCO_3$, extracting with ethyl acetate and evaporating the extract (aminophenols). No water-soluble active ingredients were found in the remaining aqueous phase. If wetting agents and softeners are present they are removed before fractionating by first extracting the acid solution with petroleum ether (if emulsion form, $CHCl_3$ or CCl_4 can be used but these must be washed with dil. NaOH to remove phenols also extracted by them). The fractions obtained are further studied by spot tests on paper. If the fraction is a mixture it is subjected to descending paper chromatography on Whatman No. 1 paper for 8–10 hr. using butanol–acetic acid–water (4:1:5) as solvent. The colourless spots are made visible by suitable reagents, e.g. $(CH_3)_2NC_6H_4CHO$ vapours, diamines turn red, amino phenols turn yellow, while phenols give purples in a stream of hot air (the colours disappearing on cooling). Ascending chromatography can also be used. Spot tests are given (with colour tables), for pyrocatechol, resorcinol, hydroquinone, and pyrogallol

(fraction 1); *o*-, *m*- and *p*-dimethylbenzene, *p*-phenylenediamine-2-sulphonic acid, 2:4-diaminoanisole, 1:2:5- and 1:2:4-methyl phenylenediamine, *p*-aminodiphenylamine, *pp*-diaminodiphenylamine and 1:8-naphthylenediamine (fraction 2); *o*-, *m*- and *p*-aminophenol, nitro-*p*-aminophenol, Amidol and Metol (fraction 3). Eleven reagents are used for the identification tests. Results are given of the analysis of 10 commercial hair dyes by this method. Three of them contained Pb salts. C.O.C.

Paper Partition Chromatography of Synthetic Dyes authorised for Colouring Foods in the Argentine
R. C. T. Puche
Rev. Asoc. bioquím. arg., **22**, 228–236 (1957): *Chem. Abs.*, **52**, 11303 (10 July 1958)

The dyes were spotted on S & S No. 0859 paper and developed with butanol saturated ink 10% HCl (I) or 0·5 ml. xylidine and 5 ml. conc., HCl in 10 ml. water (II). The following results were obtained (name of dye, R_f with solvent I, R_f with solvent II); Erythrosine (C.I. Food Red 14) 0·00, 0·60; Rose Bengal (Acid Red 93 or 94) 0·00, 0·42; Bordeaux Red (C.I. Acid Red 17) 0·07, 0·21; Ponceau Red 2R (C.I. Food Red 5) 0·18, 0·28; Orange I (C.I. Acid Orange 20) 0·22, 0·41; Tartrazine (C.I. Food Yellow 4) 0·47, 0·17, Indigotine (C.I. Food Blue 1) 0·94, 0·42; Guinea Green B (C.I. Food Green 1) 0·89, 0·50. C.O.C.

Detection of Naphthol Yellow (C.I. Food Yellow 1) and other Colouring Matter in Dijon Mustard
P. P. Legrand
Ann. Fals. et Fraudes, **51**, 5–18 (1958): *Chem. Abs.*, **52**, 12264 (25 July 1958)

Dijon Mustard frequently has a yellow colorant added to it, usually Turmeric (C.I. Natural Yellow 3) which is permitted or Naphthol Yellow (C.I. Food Yellow 1) which is not. Paper chromatography enables identification under ultraviolet radiation when Naphthol Yellow appears to be brown. C.O.C.

Quantitative Analysis of Substituted Vat Dyes
M. Matrka, F. Navrátil, and J. Filipi
Chem. Prûmysl., **7**, 343–347 (1957): *Chem. Abs.*, **52**, 12403 (25 July 1958)

A description of a potentiometric method based on the oxidation of leuco dyes by ceric sulphate, or possibly also by $K_2Cr_2O_7$ or ferric ammonium sulphate. The best results were obtained for polycyclic and anthraquinone dyes. The method is not suitable for indigoid and thioindigoid dyes as they are two readily oxidised by atmospheric O. A paper chromatographic method is described for identifying dispersing agents, stabiliser, etc. present in the commercial dye. C.O.C.

Chromatographic Study of New Dyes derived from Coal Tar allowed by the Food Authority for Use in Foods
I. Macció
Anales direc. nacl. quím. (Buenos Aires), **9**, (17), 10–12 (1956): *Chem. Abs.*, **52**, 9461 (10 June 1958)

The following dyes were separated by use of a mixture of 80% ethanol and 5% glacial acetic acid in water (R_f values after each): Erythrosine (C.I. Food Red 14) (0·88), Rose Bengal (C.I. Acid Red 93 or 94) (0·88), Orange I (C.I. Acid Orange 20) (0·76), Ponceau 2R (C.I. Food Red 5) (0·24), Naphthol Yellow S (C.I. Food Yellow 1) (0·34), Indigocarmine (C.I. Food Blue 1) (0·08), Tartrazine (C.I. Food Yellow 4) (0·07), Guinea Green B (C.I. Food Green 1) (0·90), and Patent Blue (C.I. Food Blue 3) (0·93). After separation the dyes were identified by use of the Klett–Saummerson photocolorimeter. Separation required 7 hr. in a glass vessel 10 cm. diameter × 50 cm. high. It was done on Whatman No. 1 paper 8 × 30 cm., using a 0·1% aq. soln. of dye. C.O.C.

Paper Chromatography of Organic Colorants in Food
J. M. Calvo
Anales Bromatol. (Madrid), **9**, 423–445 (1957): *Chem. Abs.*, **52**, 9461 (10 June 1958)

The aqueous ethylpyridine acetate first proposed by Jermyn and Isherwood (*Chem. Abs.*, **43**, 8312 (1949)) for chromatography of glucides is also useful for separating and identifying organic colorants in food. Sensitivity is about 0·001 mg. of colorant. C.O.C.

Detection of the Food Dye Rhodamine B (C.I. Food Red 15)
E. Beck
Pharmazie, **12**, 825–7 (1957): *Chem. Abs.*, **52**, 7552 (10 May 1958)

Detection of Rhodamine B in foods is sometimes difficult. It is better to prove its presence by fixing it on wool in ammoniacal solution rather than in acid as is the official method. Foods dyed with Rhodamine B clearly show in ultraviolet radiation a characteristic pink to yellowish orange fluorescence which is a good preliminary indication. Unlike all other food dyes permitted in E. Germany it forms a colourless solution in ether. This enables it to be separated from other food dyes, which is desirable in chromatographic analysis of a mixture of dyes. The chromatogram of Rhodamine B, unlike that of the others, is an elongated poorly defined band and in presence of other dyes may cause confusion by overlapping them. C.O.C.

Coal-tar Dyes in Red Wine
W. Saller
Mitteilungen (Klosterneuburg), *Ser. A.*, **8**, 99–107 (1958): *Chem. Abs.*, **52**, 9512 (10 June 1958)

A comparison of various ways of determining dyes in red wine. The usual methods are too sensitive to serve as a basis for rejections on the ground of adulteration. Tolerance corresponding to the colour of cotton thread dyeing with 2 mg. Bordeaux Red/l. should be allowed. Yeast cells and bacteria can adsorb dyes so that red wines may spontaneously become free from dissolved dyes. C.O.C.

Chromatographic Analysis of Ink
K. Bogusławska
Prace Inst. Celul. Papiern. (Warszawa: WPLiS), (2), 6 pp. (1957): *Polish Tech. Abs.*, **29**, (1), 157 (1958)

The best solvent used for separating was the "organic" phase of the following mixture— 50 parts *n*-butyl alcohol, 49 parts distilled water, and 1 part conc. HCl. A developing agent (2% ferric chloride) for tannin and gallic acid present in the ink was chosen. C.J.W.H.

Instrumental Ranking of Fabric Handle. V— New Tester and its Characteristics
H. Uchida, T. Takizawa, and M. Kobayashi
J. Soc. Textile Cellulose Ind. Japan, **13**, 890–892 (Dec. 1957)

A tester in which fabric handle is assessed by measuring the withdrawal resistance of the fabric through a ring hole has been constructed, and its characteristics are discussed. It has the following merits in comparison with other methods— higher reliability of the results, easier handling of the tester, easier preparation of the test piece, and more sharply evaluated results are expected. C.J.W.H.

New Method of Testing Abrasion Resistance
G. Dürig and W. Augsburger
Textil-Rund., **13**, 259–267 (May 1958)

The construction and operation of the Schiefer apparatus is described. The machine is compared with the E.M.P.A. abrader. S.R.C.

Determination of Water Vapour from the Change in Electrical Resistance of a Hygroscopic Film
E. R. Weaver, E. E. Hughes, and A. W. Diniak
Bur. Stand. J. Res., **60**, 489–508 (May 1958) *Research Paper* 2864

The electrical conductivity of a thin film of such a material as phosphoric acid changes with variation in the concentration of water in the atmosphere with which it is in contact. This is made the basis of a method of determining the amount of water vapour in a gas. Modifications of apparatus, methods, and calculations described earlier are given and applications discussed. The method is simple, rapid, and very sensitive. It requires only small samples. W.R.M.

Apparatus for the Determination of the Capillarity of Fabrics
A-N. Braslavskii
Tekstil. prom., **18**, 53–55 (May 1958)

A fairly simple apparatus for the determination of the proportion of pores in a given porous substance, based on an empirical method, is described. G.J.K.

Applications of Chromatography in Wool Research
M. Van Overbèke
Bull. Inst. Text. France, (72), 7–23 (Feb. 1958)
A review of selected applications. J.C.F.

Determination of the Homogeneity of Blends of Wool and Synthetic Fibres
G. Gianola
Bull. Inst. Text. France, (71), 77–90 (Dec. 1957)
The coefficients of mass variation of the yarn are measured before and after dissolving out the wool with 3% sodium hydroxide at 100°c. The appropriate statistical theory is given. J.C.F.

Viscosimetric Determination of Damage to Silk Fibroin
B. Rewieńska-Kościukowa
Prace Inst. Jedw. Naturaln (Warszawa: WPLiS), (1), 20 pp. (1957):
Polish Tech. Abs., **29**, (1), 137 and 139 (1958)
Experimental investigation of Korchagin's method afforded the following conclusions— (1) The viscosimetric method may be used in determining damage to silk fibres resulting from various chemical treatments. (2) A copper–ammonia solvent should be used for dissolving silk. (3) Determinations are best made 60 min. after dissolution of the silk has started. (4) In viscosimetric determinations, 1% silk solutions should be used. (5) The results can serve as a basis for working out instructions for industrial control of the production of silk. (6) The viscosimetric method is sufficiently exact to be used in analysing a number of problems in the chemical treatment of silk. C.J.W.H.

Quantitative Analysis of Wool–Terylene Blends
E. V. Truter
J.S.D.C., **74**, 303–304 (April 1958)

Measurement of Detergent Power
J. Vallée
Teintex, **23**, 321–336 (May 1958)
The behaviour of films of surface-active agents in an extensible stirrup of Pt wire has been studied. Wetting agents have short extensibilities, detergents long, with a maximum which affords a true measure of detergent power. This agrees with relative values obtained by orthodox methods on soiled materials. S.R.C.

The Limiting Redox Potentials of Vat Dyes in Solution
I. D. Zakhar'evskaya, V. A. Lentovskaya, and M. S. Zakhar'evskiĭ
Tekstil. prom., **18**, 71–72 (May 1958)
Two vat dyes, Red KKh and Golden Yellow ZhKh, have been investigated spectrophotometrically, and the optical density readings of the absorption maxima corresponding to those of the oxidised and reduced forms have been used, together with the measured redox potentials of the above system, to calculate limiting redox potentials. G.J.K.

Determination of Dye Selectivity
E. S. Yampol'skaya and I. V. Fodiman
Tekstil. prom., **18**, 28–31 (April 1958)
The affinities of insoluble azo dyes for secondary cellulose acetate (I) and Capron (6-nylon) (II) are determined by the spectrophotometric estimation of the desorbed dye using Cellosolve (for I) and by estimating the dye concn. in the residual bath (for II). G.J.K.

Modern Views on the Problem of Colour Tolerances
P. Mougeot
Teintex, **23**, 404–415 (June 1958)
A number of formulae used for measuring colour differences (Hunter, Schofield, Adams–Nickerson–Stultz, Saunderson–Milner and McAdam) are considered. The problem of tolerance is largely influenced by factors outside the field of colorimetry and depends on the degree of divergence (ΔE) admitted by producer and consumer in collaboration. S.R.C.

Dependence of Luminance required for Constant Brightness upon Chromaticity and Chromatic Adaptation
E. J. Breneman
J. Opt. Soc. Amer., **48**, 228–232 (April 1958)

Comparison of Scotopic, Mesopic, and Photopic Spectral Sensitivity Curves
J. A. S. Kinney
J. Opt. Soc. Amer., **48**, 185–190 (March 1958)
With a 2° stimulus, 10° from the fovea, scotopic characteristics are retained for 3·5–4·0 log units above absolute threshold. This sensitivity is inherent in the peripheral location and is not due to the use of the dark-adapted eye. Curves presented show a gradual increase in sensitivity toward longer wavelengths with increasing intensity but no large shift until a level 5 log units above threshold. Even then, peripheral sensitivity is lower than foveal. Essentially the same curves are obtained with dark and white light surrounds, sensitivity to longer wavelengths being slightly higher with the latter, which also much improves brightness discrimination. R.B.B.

Accuracy of Tristimulus Computations
J. L. F. De Kerf
J. Opt. Soc. Amer., **48**, 334–338 (May 1958)
Tristimulus computations were made by standard methods for twenty reflecting surfaces and filters. Only the weighted ordinate method with $\Delta\lambda = 5$ mμ. determined x, y, Y with an uncertainty smaller than $\frac{1}{2}$ just perceptible step (approx. $\frac{1}{10}$ N.B.S. unit). With $\Delta\lambda = 10$ mμ. or by the 100 selected ordinates method, x, y, Y, may be determined with an uncertainty of $\frac{1}{2}$ N.B.S. unit. Thirty selected ordinates gave uncertainties greater than $\frac{1}{2}$ N.B.S. unit. R.B.B.

Visual Selection of Colour-film Neutrals— Standard Observer
R. W. Burnham
J. Opt. Soc. Amer., **48**, 215–224 (April 1958)
Colour film selected as neutral for the C.I.E. standard observer frequently does not match a neutral for real observers, even when viewed under standard-observer conditions. Data from 29 observers show low precision of visual selection as compared with standard physical techniques, and possible deviance from C.I.E. standard observer in the blue direction. The C.I.E. standard observer was, however, more representative of the real observers than was the Stiles tentative mean observer. R.B.B.

Preparation of Colour-wedged Film Samples for Use in a Neutral Selector
R. H. Morris
J. Opt. Soc. Amer., **48**, 225–227 (April 1958)
Mathematical development of a technique for arriving at the required conditions for exposing film sheets to give equiluminous area with uniform colour gradients. R.B.B.

Colorimetric Method for Determining Dialdehyde Content of Periodate-oxidised Starch
C. S. Wise and C. L. Mehltretter
Anal. Chem., **30**, 174–175 (Feb. 1958)
In a rapid method for the determination of small percentages of carbonyl groups in periodate-oxidised starches, a *p*-nitrophenylhydrazone of the oxystarch is precipitated from aq. soln., filtered, and dissolved in ethyl alcohol, and the absorbance of the yellow soln. is measured spectrophotometrically. L.T.W.

Instruments for Brightness Measurements
E. K. Whitener and G. C. Maltenfort
Fibre Containers and Paperboard Mills, **43**, (2), 62–63, 65 (1958):
Chem. Abs., **52**, 8548 (25 May 1958)
A comparison of the Colormaster, the Photovolt and the General Electric brightness tester. The Colormaster is preferred for controlling production because it is easily calibrated, views a large area of paper and its reading can be accurately, quickly and reliably converted to G.E. brightness by the equation G.E. brightness = 0·958 × Colormaster brightness + 2·136. The relationship between Photovolt and G.E. brightness is G.E. brightness = 1·137 × Photovolt brightness − 12·561 for Photovolt readings of 51·5 – 66·0 and G.E. brightness = 1·321 × Photovolt brightness − 25·551 for Photovolt reading of 66·0 – 81·0. C.O.C.

Paperboard and Ink Colour Measurements
G. G. Maltenfort and E. K. Whitener
Fibre Containers, **43**, 94–97, 100 (May 1958)
By simple regression analysis the readings of the Colormaster (a tristimulus instrument) can be converted into

G.E. spectrophotometer reflectance readings. Earlier work (*ibid.*, **43**, 62, 63, 65 (Feb. 1958)) showed that Colormaster blue filter readings show a nearly perfect correlation with G.E. brightness. It is now shown that there is nearly perfect correlation between Colormaster blue, green and red filter readings and G.E. spectrophotometer reflectances. R.A.

Determination of Fastness to Weathering
E. Brunnschweiler
Textil-Rund., **13**, 226–231 (April 1958)

An account of work carried out on standardisation by a committee of the Swiss Dye Fastness Commission in various countries, together with recommendations. S.R.C.

Assessment of Fastness to Perspiration
M. Bühler
Melliand Textilber., **39**, 285–288 (March 1958)

Laboratory test methods are critically reviewed. Reference is made to the removal of copper from metallised dyes by certain constituents of perspiration such as amino- and hydroxy-carboxylic acids. L.A.T.

Reliability of the Soil Burial Method for evaluating Textile Preservative Treatments
J. M. Ashcroft
Text. Research J., **28**, 422–430 (May 1958)

The variability in the degradation rate of a fabric treated with each of two rot-inhibiting treatments was measured after burial in a biologically active soil for varying periods of time under controlled conditions. The experiment enabled 10 observations to be made in each of 5 soil blocks for each measurement. The results showed that variability in fabric strength measurements increased as deterioration progressed, being greatest for the treated fabrics. However, the rate of deterioration of the untreated fabrics in different soil blocks showed marked variability, an effect not noticed in most measurements on the treated fabric. The shortcomings inherent in the general practice of reporting the results of soil exposure tests as the average % of strength lost or retained are discussed and a new method of reporting data is suggested. S.B.D.

Brightness of Bleached Pulp—Tentative Standard CCA 27:58
Central Laboratory of the Cellulose Industry
Svensk Papperstidning, **61**, 358–359 (15 June 1958)

Brightness is determined as the reflectance of diffused light of wavelength 457 mμ. compared with the reflectance of magnesium oxide, taken as 100%. The apparatus used is the Elrepho reflection meter, with R46 TAPPI filter, calibrated so that laboratory sheets prepared according to CCA 26:55 give the same brightness values as the master instrument, TAPPI 217m-48. Normally four similar sheets are placed under each other, but a minimum of two sheets may be sufficient, provided they are backed with sheets having a brightness which differs by < 5 units from that of the sample. R.A.

Complexometric Determination of Copper in Cupriethylenediamine Solutions
J. Šára and W. Berndt
Svensk Papperstidning, **61**, 353–355 (15 June 1958)

Approx. M. cupriethylenediamine soln. (25 ml.) is diluted to 250 ml. and shaken well. A few drops of Dimethyl Yellow or Methyl Orange are added to 5 ml. in a 500-ml. flask, and the mixture is titrated to red with N-HCl. Exactly 15 ml. of a 0·05 M. Complexone III soln. is added, the mixture made up to approx. 350 ml., and 10 ml. of a 20% urotropine soln. (or 2 g. solid urotropine) and a few drops of Xylenol Orange (1% aq. soln.) or approx. 0·2 g. Methylthymol Blue (mixed with solid KNO_3 in the ratio 1 : 100) are added. Excess Complexone is back-titrated with a 0·05 M. Pb or Zn salt soln. The molarity is given by—

$$[Cu] = (a-b) \times 0{\cdot}1 \text{ M.}$$

where a = vol. of 0·05 M-Complexone III soln. in ml. and b = vol. of 0·05 M. Pb or Zn soln. used in ml. The Complexone is standardised against pure lead chloride, and then used to standardise the lead or zinc soln. Alternatively, the latter are prepared as standard soln. from lead nitrate dried at 180°C. or zinc oxide ignited at 300°C., and the Complexone is standardised against these. All titrations are carried out at pH 5 (with urotropine). R.A.

Colorimetric Estimation of Crystal Violet Base (C.I. Solvent Violet 9) taken up by the Acidic Groups of Cellulose
M. Rebek and H. Beck
Das Papier, **12**, 201–203 (May 1958)

The Crystal Violet method has the advantage over the Kjeldahl determination that much smaller samples are needed. Acidity determinations can be made with 0·1–0·05 g. in half the time and 10% of the amount of solvent. The reproducibility and the sensitivity of the method are illustrated by examples for cotton and sulphite pulp. R.A.

Molecular Weight Distribution of Cellulose. II—Rapid Determination of Molecular Weight Distribution by the Precipitation Method
K. Yagami and T. Kanō
J. Soc. Textile Cellulose Ind. Japan, **13**, 837–841 (Dec. 1957)

The standing time for precipitation was decreased, and the fractionation was carried out in a small volume of solution (0·1–0·5 g. samples and 200 ml. solvent, instead of 3–5 g. samples and 1 litre solvent). This procedure gave similar distribution curve and reproducibility to the ordinary method, although only one-fifth of the time of the ordinary method was required. The maximum in the distribution curve was shifted towards higher molecular weights compared with the ordinary method. The uniformity of the first fraction depends on the standing time allowed for precipitation. The first fraction which is obtained in the minimum standing time contains less low-mol.wt. component than that in 45 hr. The precipitate will absorb selectively the low-mol.wt. component and will be depolymerised, when the procedure requires a longer time. Fractionation from a 0·25% solution was slightly sharper than that from a 0·5% solution. C.J.W.H.

Brightness Measurements on Pulp Suspensions
T. Ploetz, R. Langer, and H. Meyer
Das Papier., **12**, 368–371 (July 1958)

For mill control the brightness of a paper can be calculated with sufficient accuracy from simple brightness tests applied to the pulp suspensions. The Brecht–Liebert apparatus was not found to be sufficiently accurate. A Zeiss electrical remission photometer, Elrepho, which is generally used for measuring the degree of remission of solid substances and powders, was found satisfactory. The sample was illuminated by diffused light over an Ulbricht sphere, and the light reflected upwards from the sample was measured using colour filters. By using this method the time-consuming formation of test sheets is avoided, unskilled labour can carry out the tests, and in the bleaching plant immediate correction can be made for variations in brightness. R.A.

Determination of the Degree of Polymerisation of Cellulose from Viscosity Data in Benzyltriethylammonium Hydroxide
N. S. Nikolaeva, E. M. Mogilevskiĭ, and Z. K. Lin'kova
Tekstil. prom., **18**, 9–11 (April 1958)

Values obtained for the D.P. of cellulose from viscosity data using solutions of various cellulosic materials in benzyltriethylammonium hydroxide (I) compare favourably with those derived from viscosity measurements in cuprammonium hydroxide solutions. The constant K_m (Staudinger's equation) of I is given as $5{\cdot}7 \times 10^{-4}$, and this constant remains unchanged for about 48 hr. even without an inert atmosphere. G.J.K.

High-speed, Direct-reading Glossmeter
J. G. Schreckendgust and D. M. Gowing
J. Opt. Soc. Amer., **48**, 241–245 (April 1958)

Determination of α-Glycol Content of Epoxy Resins
G. A. Stenmark
Anal. Chem., **30**, 381–383 (March 1958)

The α-glycol content of epoxy resins is determined by treatment with a quaternary ammonium periodate in chloroform as solvent. The excess periodate is determined iodometrically. The reagent used is benzyltrimethylammonium periodate; the method gives accurate results with pure glycols and is sensitive to small amounts of glycol groups in epoxy resins. L.T.W.

Assessment of the Colour Fastness of Leathers

H. E. Nursten

J. Soc. Leather Trades' Chem., **42**, 166–171 (May 1958)

Methods for determining the fastness of coloured leathers to water, washing, perspiration, organic solvents, daylight, rubbing, heat, and marking off, are described; they have been established by the Fastness Tests Committee of the Society of Leather Trades' Chemists. The Grey Scale is used as the basis of evaluation in every case, though the Blue Scale (*B.S.* 1006:1955) is used in determining light fastness. It is stressed that these are tests of the fastness of coloured leathers and not of the colorants as such. J.W.D.

Provisional Method for testing Fastness (of Leather Finishes) to Water Spotting

Society of Leather Trades' Chemists Fastness Test Committee

J. Soc. Leather Trades' Chem., **42**, 231–232 (July 1958)

Two drops of distilled water are placed on the leather sample; after 30 min. surplus water is removed from one of the drops with filter paper, and any physical effects are observed. The other drop is allowed to evaporate overnight; the change in colour of the leather is then assessed with the standard Grey Scale. J.W.D.

Provisional Method for testing the Colour Fastness of Leather Finishes to Heat

Society of Leather Trades' Chemists Fastness Test Committee

J. Soc. Leather Trades' Chem., **42**, 226–229 (July 1958)

The leather, previously conditioned in a standard atmosphere, is placed in contact with the flat face of a heated metal member of specified dimensions for a period of 5 sec. with a contact pressure of 3 lb. per sq.in. After cooling the resultant change of colour is assessed with the standard Grey Scale, and other changes in appearance of the finish are noted. Tests are carried out at 150°C., 200°C., and 250°C. The apparatus is available commercially and is shown in a diagram. J.W.D.

Provisional Method for Determining the Transfer of Colour (of Leather Finishes) in Washing

Society of Leather Trades' Chemists Fastness Test Committee

J. Soc. Leather Trades' Chem., **42**, 229–231 (July 1958)

The sample of coloured leather is sewn between two pieces of the relevant textile material and washed five times in soap soln. of specified strength, rinsing after each wash. After drying the suspended composite specimen at 60°C., the staining of the textile material is assessed by the standard Grey Scale. J.W.D.

Applications of Infrared Absorption Spectroscopy to Investigations of Cotton and Modified Cottons. II— Chemical Modifications

R. T. O'Connor, E. F. DuPré, and E. R. McCall

Text. Research J., **28**, 542–554 (July 1958)

Application of the KBr disc technique has demonstrated that chemical modification can be readily detected and identified and that the extent of treatment can be quantitatively estimated. Experiments have been designed to select bands most useful in investigations of cotton modified by esterification, etherification, or by replacement reactions. A list of 50 bands and the vibrating groups giving rise to them is presented, which summarises the spectral characteristics which have been found most useful in the investigations of cottons modified in the above ways. S.B.D.

Simple Device for the Measurement of the Crease-recovery Angle

M. Menke

Melliand Textilber., **39**, 552–554 (May 1958)

Dry wrinkle-recovery angles vary appreciably after repeated washing at the boil, but the wet values remain substantially unaltered. The Monsanto tester is suitable, and so is the official method adopted for the quality mark Rapid Iron, but as the protractor in this method has to be held manually, fatigue and inaccuracy can arise. This is obviated in the Pfersee instrument illustrated, in which a graduated transparent semicircular basal plate is grooved to receive a sample carrier after the removal of load. Dry wrinkle-recovery angles can also be measured. L.A.T.

Assessment of Wash-and-wear Characteristics of Fabrics

B. S. Marek

S.V.F. Fachorgan, **13**, 247–257 (May 1958)

After reviewing existing procedures, the author describes the development and application of a new method for the assessment of retention of wash-and-wear characteristics of resin-treated fabrics. Assessments of 28 different fabrics, based on the newly developed system of rating on the 1–5 scale, are tabulated, discussed, and compared with the Monsanto method of measuring the crease-recovery angle in the dry and the wet state. L.A.T.

PATENT

Abrasion Tester for Fabric, etc.

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A machine which enables the fabric under test to be kept in a relaxed condition. C.O.C.

Indicator Dyes (IV p. 713)

Application of "Chromatoelectrophoresis" to the Analysis of various Morphological Fractions of Wool (VI p. 716)

Dyeing Behaviour of South African Wools (VI p. 716)

Comparative Mechanical Properties of Cotton Fibres and Primary Flax Cells— Effect of Degradation by Hypochlorites (VII p. 717)

Colour Reaction for Non-ionic and Amphoteric Soaps (VIII p. 718)

Investigation of the Dyeing of Oxidised Cellulose by Infrared Spectroscopy (VIII p. 718)

Finishing of Textiles with Preserving and Proofing Agents and their Testing against Cellulose-attacking Micro-organisms (A Note on the DIN Proposals No. 53930 and 53933) (X p. 721)

Pilling and its Prevention (X p. 721)

XV— MISCELLANEOUS

PATENTS

Wood Stain containing Pigments

Interchemical Corpn. *USP* 2,802,712

A solution of a pigment, e.g. the phosphotungstic, phosphomolybdic or tannic acid salt of a basic dye, in formamide or a liquid of formula R^1-CO-NR^1-R^2 (R^1 = H or Alk; R^2 = H, Alk, OH or alkoxy-substituted Alk), e.g. dimethylformamide, gives rapid staining of wood. The stained wood is brilliantly coloured with even penetration. C.O.C.

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USP 2,805,914

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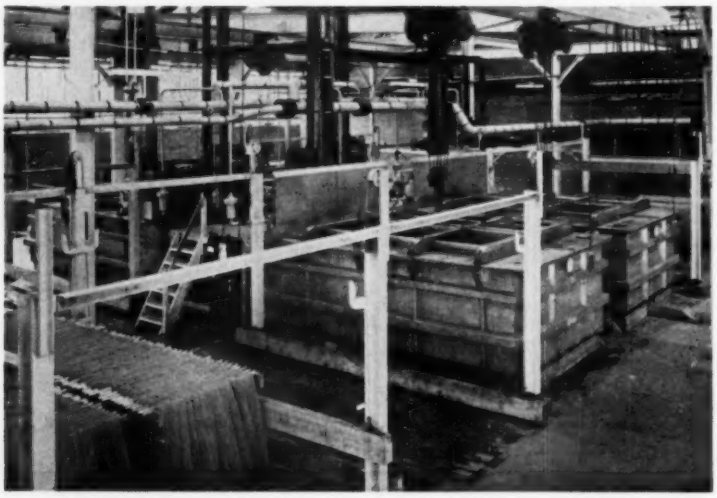

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FORTHCOMING MEETINGS OF THE SOCIETY—*continued from page viii*

Thursday, 11th December 1958

WEST RIDING SECTION. *The Development of a Dyestuff from its Synthesis to its Marketing.* K. Roessler, Esq. (Badische Anilin & Soda-Fabrik). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Tuesday, 16th December 1958

HUDDERSFIELD SECTION. *The Development of a Dyestuff from its Synthesis to its Marketing.* K. Roessler, Esq. (Badische Anilin & Soda-Fabrik A.G., Germany). (Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, **Huddersfield.** 7.30 p.m.

Thursday, 18th December 1958

MANCHESTER JUNIOR BRANCH. *Modern Package Dyeing.* A. Gibb, Esq. Room J/B 16—Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 19th December 1958

MANCHESTER SECTION. *Felisol— An International Guarantee of Colour Fastness.* K. McLaren, Esq., B.Sc., F.R.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Tuesday, 6th January 1959

LONDON SECTION. *To Blend or not to Blend—That is the Question.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Leeds University). (Joint meeting with the London Section of the Textile Institute.) Chemical Society, Burlington House, **London W.1.** 6.30 p.m.

Thursday, 8th January 1959

WEST RIDING SECTION. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 9th January 1959

LONDON SECTION. *Optical Brightening Agents in Action.* J. L. Ashworth, Esq., B.Sc., Ph.D., H. B. Mann, Esq., T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C., F.T.I. (Courtaulds Ltd., Textile Research Laboratory, Bocking.) The Royal Society, Burlington House, **London, W.1.** 6 p.m.

Tuesday, 13th January 1959

NORTHERN IRELAND SECTION. *Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials.* F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *Dyeing Unions Containing Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Thursday, 15th January 1959

MANCHESTER JUNIOR BRANCH. *Management.* Prof. R. W. Revans. Room J/B 16—Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 16th January 1959

MANCHESTER SECTION. *The Degradation of Chemically Modified Celluloses by Alkali.* Dr. W. M. Corbett (British Rayon Research Association). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Monday, 19th January 1959

HUDDERSFIELD SECTION. Title to be announced later. Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I., F.S.D.C. (Joint meeting with the Huddersfield Textile Society.) Large Hall, Technical College, **Huddersfield.** 7.30 p.m.

Wednesday, 21st January 1959

MIDLANDS SECTION. *The Solubility of Wool Dyes . . . Assessment and Practical Significance.* W. Beal, Esq., B.Sc. College of Technology, **Leicester.** 7 p.m.

Thursday, 22nd January 1959

WEST RIDING SECTION. *Some Aspects of Reactive Dyes.* Dr. J. Wegmann. (Ciba Ltd., Basle). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 23rd January 1959

LONDON SECTION. Annual Dinner and Dance. Waldorf Hotel, **London, W.C.2.** 7 p.m. for 7.30 p.m.

Friday, 23rd January 1959

WEST RIDING SECTION. Ladies Evening. Victoria Hotel, Bridge Street, **Bradford.**

Monday, 26th January 1959

BRADFORD JUNIOR BRANCH. *Recent Developments in the use of Terylene Staple Fibre in the Woollen and Worsted Industry.* P. W. Eggleston, Esq. (Imperial Chemical Industries Ltd.). Institute of Technology, **Bradford** 7.15 p.m.

Saturday, 31st January 1959

MANCHESTER SECTION. Annual Dinner and Dance, Grand Hotel, **Manchester.** 6 p.m. for 6.30 p.m.

Tuesday, 3rd February 1959

LEEDS JUNIOR BRANCH. *Colour Matching.* E. Bellhouse, Esq., B.Sc. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

Thursday, 5th February 1959

MIDLANDS SECTION. *Developments in Modern Dry-cleaning Techniques.* E. J. Davies, Esq., M.Sc. Gas Board Theatre, **Nottingham.** 7 p.m.

Friday, 6th February 1959

LONDON SECTION. *Colour Photography.* Dr. R. W. G. Hunt (Kodak Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

Tuesday, 10th February 1959

BRADFORD JUNIOR BRANCH. *New Levelling Agents in Acid and Direct Dyebaths.* G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

NORTHERN IRELAND SECTION. *Emulsion Thickenings—Possibilities in Textile Printing.* T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. *The Use of Antistatic Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). (Joint meeting with the Textile Institute.) St. Enoch Hotel, **Glasgow.** 7.15 p.m.

Thursday, 12th February 1959

WEST RIDING SECTION. *New Ideas in the Application of Phthalocyanine Derivatives to Textiles.* Dr. F. Gund. (Farbenfabriken Bayer). Griffin Hotel, Boar Lane, **Leeds.** 7.30 p.m.

Tuesday, 17th February 1959

HUDDERSFIELD SECTION. *The Use of Antistatic Agents in Textile Processing—Advantages and Disadvantages.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.) Silvios Cafe, **Huddersfield.** 7.30 p.m.

Thursday, 19th February 1959

MANCHESTER JUNIOR BRANCH. *Chlorite Bleaching.* W. J. Watts, Esq., B.Sc. Room J/B 16—Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 20th February 1959

MANCHESTER SECTION. *Emulsion Thickenings. Possibilities in Textile Printing.* T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C., F.C.S. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, **Manchester.** 7 p.m.

Wednesday, 25th February 1959

BRADFORD JUNIOR BRANCH. *Vat Dyeing of Cotton Piece Goods by Pre-pigmentation Routes.* M. R. Fox, Esq., F.T.I., F.S.D.C. and J. F. Mawson, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

Thursday, 26th February 1959

WEST RIDING SECTION. *Fast Shades on Wool–Cellulose Unions.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 6th March 1959

LONDON SECTION. *The Use of Antistatic Agents in Textile Processing.* A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). The Royal Society, Burlington House, **London W.1.** 6 p.m.

MIDLANDS SECTION. Section Annual Dinner. George Hotel, **Nottingham.**

continued on page xlii

FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page* xli

Tuesday, 10th March 1959

LEEDS JUNIOR BRANCH. *New Methods for the Assessment of Dyeing Properties and Usability of Dyes.* G. H. Lister, Esq., B.Sc., Ph.D. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds.** 3.15 p.m.

NORTHERN IRELAND SECTION. *Stenter Drying.* T. A. Uthwatt, Esq., B.A., A.M.I.E.E., and J. S. Woollatt, Esq., B.Sc. (Mather & Platt Ltd.). Grosvenor Rooms, **Belfast.** 7.30 p.m.

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by *Modern Cloth Drying Principles and Machines.* K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). St. Enoch Hotel, **Glasgow.** 7·15 p.m.

Thursday, 12th March 1959

BRADFORD JUNIOR BRANCH. *Sizing Materials— Their Chemistry and Application.* J. H. MacGregor, Esq., Ph.D., F.R.I.C., F.C.S. (Courtaulds Ltd.). Institute of Technology, **Bradford.** 7.15 p.m.

MIDLANDS SECTION. *Dyeing and the Future.* J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C., President of the Society. (Joint meeting with the Coventry Textile Society.) Courtauld's Acetate and Synthetic Fibres Laboratory, Lockhurst Lane, **Coventry.** 7 p.m.

SCOTTISH SECTION. *The Reactivity of Keratin.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I., F.S.D.C. Scottish Woollen Technical College, **Galashiels.** 7.30 p.m.

WEST RIDING SECTION. *The Sorption of Moisture by Fibre-forming Polymers.* L. Valentine, Esq., B.Sc., Ph.D. (Tootal Broadhurst Lee Co. Ltd.). Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Saturday, 14th March 1959

BRADFORD JUNIOR BRANCH. Annual General Meeting. Institute of Technology, **Bradford.** 10.15 a.m.

Tuesday, 17th March 1959

HUDDERSFIELD SECTION. *Fast Shades on Wool-Cellulose Unions. Recent Advances in this Field.* D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Wednesday, 18th March 1959

MIDLANDS SECTION. *Photoelectric Colorimeters . . . their uses and limitations in the Dyehouse.* J. V. Alderson, Esq., B.Sc., A.R.C.S. (Joint meeting with the British Association of Chemists.) Midland Hotel, **Derby.** 7 p.m.

Thursday, 19th March 1959

MANCHESTER JUNIOR BRANCH. Films— *Silk. Principles of Chromatography. How the World Looks to a Colour Defective.* Room J/B 16 — Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 20th March 1959

MANCHESTER SECTION. *Symposium on Re-active Dyes.* College of Science and Technology, **Manchester.** Further details later.

Thursday, 2nd April 1959

MIDLANDS SECTION. *Colour Physics and Match Prediction.* (Lecturer to be announced later.) (Joint meeting with the Textile Institute.) Carpet Trades Ltd. Canteen, **Kidderminster.** 7 p.m.

Friday, 3rd April 1959

LONDON SECTION. *Recent Developments in Dyeing.* T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Imperial Chemical Industries Ltd.). George Hotel, **Luton.** 7 p.m.

Thursday, 9th April 1959

WEST RIDING SECTION. Annual General Meeting. Colour Film on *How the World Looks to a Colour-defective.* Victoria Hotel, Bridge Street, **Bradford. 1**

Friday, 10th April 1959

The Society's Annual General Meeting and Dinner, Grand Hotel, **Leicester.**

Wednesday, 15th April 1959

MIDLANDS SECTION. Annual General Meeting of the Section followed by a showing of New Scientific Films. King's Head Hotel, **Loughborough.** 7 p.m.

Thursday, 16th April 1959

MANCHESTER JUNIOR BRANCH. Three short papers by members of the section. Room J/B 16 — Chemical Engineering Building (Jackson Street), College of Science and Technology, **Manchester.** 4.30 p.m.

Friday, 17th April 1959

MANCHESTER SECTION. Annual General Meeting. *Stenter Drying.* A. T. Uthwatt, Esq. (Mather & Platt Ltd., Research Dept.). The Textile Institute, **Manchester.** 7 p.m.

Tuesday, 21st April 1959

HUDDERSFIELD SECTION. Annual General Meeting. Followed by a Colour Film on *How the World Looks to a Colour-defective.* Cmdr. Dean Farnworth (United States Naval Research). Silvios Cafe, **Huddersfield.** 7.30 p.m.

Friday, 24th April 1959

LONDON SECTION. Annual General Meeting. Commander Dean Farnworth (United States Navy) will show and discuss his film *How the World Looks to a Colour-defective.* Waldorf Hotel, **London W.C.2.** 6 p.m.

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xlvii	Samuel Bradley Ltd
xx	Brotherton & Co Ltd
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xxi	Catomance Ltd
xviii	Ciba Ltd
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xv	Cochran & Co Annan Ltd
xlviii	Cole & Wilson Ltd
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xlii	Chas Forth & Son Ltd
xxxviii	Geigy Co Ltd
xlv	Hardman & Holden Ltd
xxx	Hickson & Welch Ltd
xxxi	L B Holliday & Co Ltd
xiv	Hunt & Moscrop Ltd
x	Imperial Chemical Industries Ltd
xxxiv	Imperial Chemical Industries Ltd
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